

The Journal *of the* SOCIETY OF DYERS AND COLOURISTS

Volume 71 Number 11

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THE SOCIETY OF DYERS AND COLOURISTS
19 PICCADILLY • BRADFORD 1 • YORKSHIRE

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Manuscripts submitted for publication in the *Journal* should be typewritten (double spacing) on good-quality paper, using one side of the paper only and leaving a margin at least 1 in. wide on the left-hand side. The time taken in refereeing papers (both lectures and communications) will be reduced to a minimum when authors submit two copies of the typescript.

In view of the high costs of publication, it is essential that authors should be as concise as possible. When experimental procedure has already been published, a literature reference to the paper containing the details is sufficient, whilst well known experimental methods should be described very briefly.

Introductory paragraphs describing the aims of the investigation and the method of attack are desirable, and should be followed by the experimental results and their discussion. There should be a brief summary for insertion at the beginning of the paper. References to the literature should be numbered consecutively, using superscript numbers without brackets immediately following the text words or author's name to which they refer.

The list of references should be given at the end of the manuscript and the abbreviations used should be, as far as possible, those given in the "List of Periodicals Abstracted" included at the end of the index to the preceding year's *Journal*. Reference numbers in this list should be neither enclosed in brackets nor followed by full-stops. As far as possible throughout the manuscript the abbreviations listed in the Jan. 1950 issue (p. 54) should be used. Tables should be numbered consecutively in Roman numerals and figures in Arabic numerals.

The number of figures and graphs should be kept as low as possible, and data should be presented in the form of either tables or graphs, not both. Drawings should be carefully prepared, preferably in Indian ink, on plain white drawing paper or, preferably, Bristol board. In graphs, the frame and actual curves should be ruled and inked more heavily than any co-ordinate lines, and the latter should not be close together as in ordinary graph paper. Experimental points should always be given, and where several graphs appear in a single figure clear means of differentiation must be adopted. All numbers and legends are set up in type by the printer, and authors should therefore indicate them lightly in pencil.

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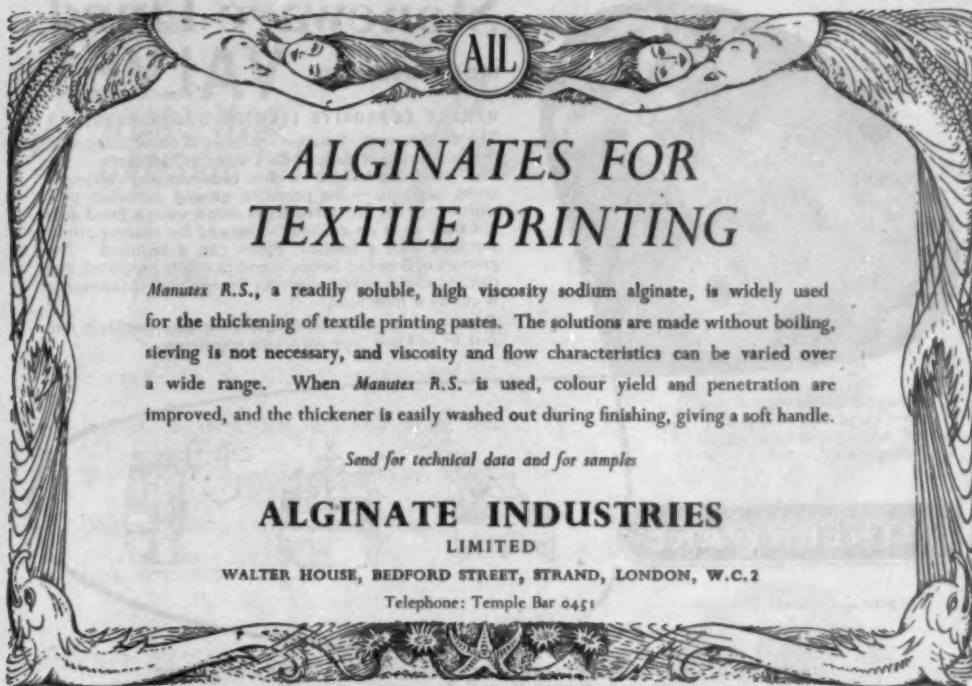
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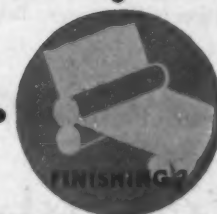
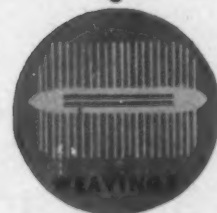
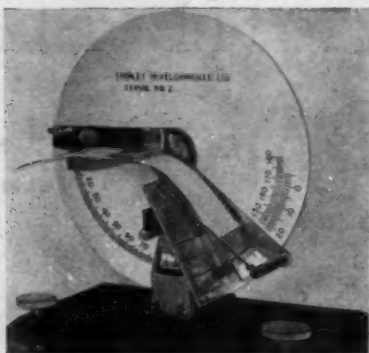
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Readers requiring general information regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1-6 of the January 1955 and pages 369-373 of the July 1955 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (Telephone Bradford 25138-9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal* —

LECTURES

Some Observations on American Bleaching and Dyeing Processes

J. K. Skelly

Estimation of Fluorescent Brightening Agents

G. G. Taylor

F.T.C.C. PUBLICATION

The Spectral Regions of Daylight which cause Fading

K. McLaren

COMMUNICATIONS

The Polarography of Azo Dyes

J. de O. Cabral and H. A. Turner

The Combination of Wool with Acids

B. Olofsson

The Irgalan Dyes — Neutral-dyeing Metal-complex Dyes

G. Schetty

EXPLANATORY PAPER ON MODERN THEORY

An Introduction to the Theory of Wool Dyeing

II—The Kinetic Approach

L. Peters

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FORTHCOMING MEETINGS OF THE SOCIETY

Wednesday, 23rd November 1955

MIDLANDS SECTION. *Modern Methods of Finishing Open-width Knitted Fabrics.* Film Evening introduced by J. Dalglish, Esq. Technical College, Nottingham. 7 p.m.

BRADFORD JUNIOR BRANCH. *Properties of Terylene/Wool Blends in Yarn and Fabric Form.* B. I. Brooke, Esq. (I.C.I. 'Terylene' Council). Technical College, Bradford. 7.15 p.m.

Thursday, 24th November 1955

WEST RIDING SECTION. *The Newer Developments in the Field of Sulphur Dyestuffs.* H. Musshoff, Esq. (Cassella Farbwerke Mainkur A.G.). The Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Friday, 2nd December 1955

LONDON SECTION. *The Application and Evaluation of Optical Bleaching Agents.* G. R. Perdue, Esq. (British Launderers Research Association). The Royal Society Rooms, Burlington House, Piccadilly, London, W.1. 6 p.m.

Tuesday, 6th December 1955

LEEDS JUNIOR BRANCH. *The Printing of Acetate Rayon.* W. B. Miller, Esq. (Messrs. British Celanese Ltd.). The Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, Leeds 2. 3.30 p.m.

Wednesday, 7th December 1955

MIDLANDS SECTION. *The Dyeing and Finishing of 'Courplets' Knitted Fabrics.* S. N. Bradshaw, Esq. Bell Hotel, Leicester. 7 p.m.

Thursday, 8th December 1955

WEST RIDING SECTION. *An Outline of the History of the Finishing of Wool Cloths.* C. O. Clark, Esq., F.T.I., F.S.D.C., Chairman, Preservation of Historical Records Committee. The Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Friday, 9th December 1955

MANCHESTER SECTION. *The Causes of Felting in Rayon Textiles.* H. W. Best-Gordon, Esq., F.C.I. The Textile Institute, 10 Blackfriars Street, Manchester 10. 6.30 p.m.

Tuesday, 13th December 1955

SCOTTISH SECTION. *Basic Mechanism in Jigger Dyeing of Woven Fabrics.* Dr. J. Ashworth (Courtaulds Ltd.). St. Enoch's Hotel, Glasgow. 7 p.m.

Wednesday, 14th December 1955

NORTHERN IRELAND SECTION. *Recent Developments in Bleaching, Dyeing and Finishing Machinery.* K. Laurie, Esq. (John Dalglish & Co. Ltd.). Thompson's Restaurant, Donegall Place, Belfast. 7.30 p.m.

Thursday, 15th December 1955

BRADFORD JUNIOR BRANCH. *Experience in the Application of Automatic Regulation to the Dyeing Process.* F. Blezard, Esq. (Drayton Regulator & Instrument Co. Ltd.). Technical College, Bradford. 7.15 p.m.

Tuesday, 20th December 1955

HUDDERSFIELD SECTION. *The Dyeing of 'Courplets'.* W. R. Beath, Esq. and J. S. Ward Esq. (Courtaulds Ltd.). Co-operative Cafe, Huddersfield. 7.30 p.m.

Wednesday, 11th January 1956

NORTHERN IRELAND SECTION. *Causes of Felting in Rayon Textiles.* H. W. Best-Gordon, Esq. (Courtaulds Ltd.). (Joint Meeting with the Textile Institute), Thompson's Restaurant, Donegall Place, Belfast. 7.30 p.m.

Thursday, 12th January 1956

WEST RIDING SECTION. *User Dyer Relations.* I. Glasman, Esq., A.T.I. (Marks & Spencer Limited). The Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Friday, 13th January 1956

BRADFORD JUNIOR BRANCH. Lecture Title to be announced later. Midland Silicones Ltd. Technical College, Bradford. 7.15 p.m.

LONDON SECTION. *The Use of Old Documents in Modern Printing.* Ronald Baker, Esq. (Swainslands Fabric Printing Company). Royal Society Rooms, Burlington House, Piccadilly, London, W.1. 6 p.m.

Tuesday, 17th January 1956

HUDDERSFIELD SECTION. Tide later. Professor J. B. Speakman.

SCOTTISH SECTION. *What Dyeing does to Wool.* F. F. Elsworth, Esq., Ph.D., A.R.I.C., F.S.D.C. (Wool Industries Research Association). St. Enoch's Hotel, Glasgow. 7 p.m.

Wednesday, 18th January 1956

MIDLANDS SECTION. Discussion Group on Detergency. Questions invited: G. H. Rostron, Esq., A.R.I.C. (Soap), R. C. Tarring, Esq., B.Sc., A.R.C.S. (Synthetics), and R. S. Hartley, Esq., A.R.I.C. (Research). Kings Head Hotel, Loughborough. 7 p.m.

Friday, 20th January 1956

MANCHESTER SECTION. *The Finishing of 'Terylene' Polyester Fibre Staple and Filament Fabrics.* G. D. Myers, Esq., B.Sc.Tech. Textile Institute, 10 Blackfriars Street, Manchester 10. 6.30 p.m.

LONDON SECTION. Perkin Centenary Dinner Dance. Waldorf Hotel, London, W.C.2. 7 p.m.

WEST RIDING SECTION. Ladies' Evening. The Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Tuesday, 24th January 1956

LEEDS JUNIOR BRANCH. *Fading—Its Nature and Assessment.* H. B. Mann, Esq. (Messrs. Courtaulds Ltd.). The Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, Leeds 2. 3.30 p.m.

Thursday, 26th January 1956

WEST RIDING SECTION. *Textile Invention and the Dry Cleaner.* A. J. Crockett, Esq., M.Sc., F.R.I.C. (Messrs. Eastmans). The Hotel Metropole, King Street, Leeds. 7.30 p.m.

Monday, 30th January 1956

BRADFORD JUNIOR BRANCH. *A New Approach to High Temperature Dyeing.* S. N. Bradshaw, Esq. (Courtaulds Coventry Ltd.). Technical College, Bradford. 7.15 p.m.

Thursday, 2nd February 1956

SCOTTISH SECTION. *Scouring and Milling.* B. F. J. Moxon, Esq. (Wool Industries Research Association). The Scottish Woollen Technical College, Galashiels. 7 p.m.

Friday, 3rd February 1956

LONDON SECTION. *A New Approach to High Temperature Dyeing.* S. N. Bradshaw, Esq. (Courtaulds Ltd., Coventry). Royal Society Rooms, Burlington House, Piccadilly, London, W.1. 6 p.m.

Tuesday, 7th February 1956

LEEDS JUNIOR BRANCH. *A New Approach to High Temperature Dyeing.* S. N. Bradshaw, Esq. (Messrs. Courtaulds Ltd.). The Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, Leeds 2. 3.30 p.m.

Wednesday, 8th February 1956

NORTHERN IRELAND SECTION. *General Principles in Vat Dyeing.* J. W. Reidy, Esq. (Messrs. L. B. Holliday & Co. Ltd.). Thompson's Restaurant, Donegall Place, Belfast. 7.30 p.m.

Thursday, 9th February 1956

WEST RIDING SECTION. *The Dyeing and Finishing of 'Terylene' and 'Terylene'/Wool Blended Fabrics.* H. R. Hadfield, Esq., M.Sc.Tech. and G. D. Myers, Esq., M.Sc.Tech. (Imperial Chemical Industries Limited, Dyestuffs Div., and Terylene Council). The Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

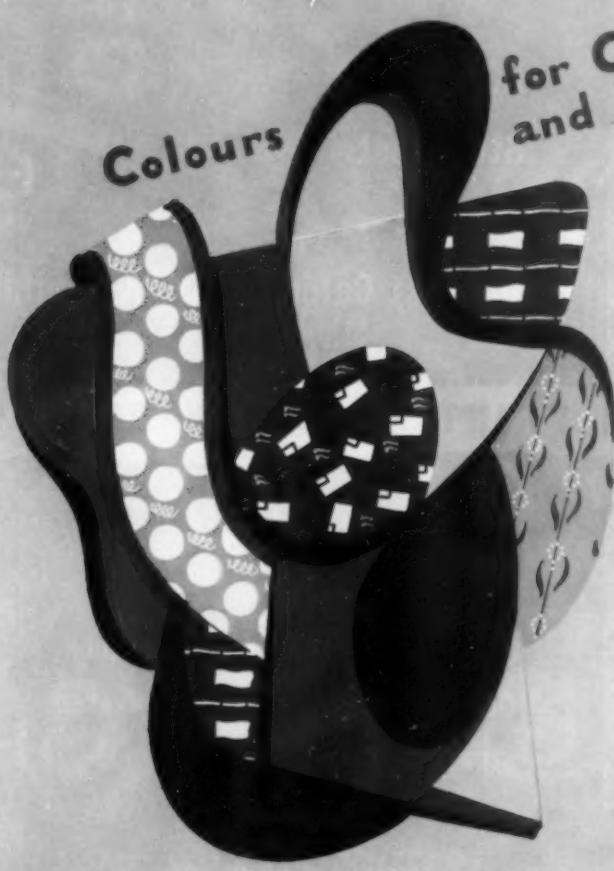
Tuesday, 14th February 1956

SCOTTISH SECTION. *The Dyeing and Finishing of 'Terylene' and 'Terylene' Blends.* J. G. Graham, Esq., and Dr. P. T. Standing (I.C.I. 'Terylene' Council). St. Enoch's Hotel, Glasgow. 7 p.m.

Wednesday, 15th February 1956


BRADFORD JUNIOR BRANCH. *Piece Scouring.* B. F. J. Moxon, Esq. (Wool Industries Research Association). Technical College, Bradford. 7.15 p.m.

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THE JOURNAL

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Volume 71 Number 11

NOVEMBER 1955

Issued Monthly

Proceedings of the Society

The Use of Silicones in the Textile Industry

T. W. WATSON

*Meeting of the Scottish Section held at the St. Enoch Hotel, Glasgow, on 16th November 1954,
Mr. R. A. Peel in the chair*

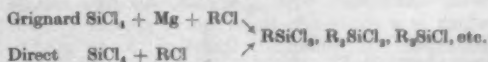
The chemistry of the production of silicones is outlined, and their main uses in the textile industry are discussed. They are mostly employed as water-repellent finishes, but their application to the shrink-proofing of wool is under investigation. Other uses are as anti-foaming agents, as release agents for sized yarns, as lubricating greases in textile machinery and for valves and stopcocks in pipelines conveying steam or corrosive chemicals, as sewing thread lubricants, and as insulators in electrical motors.

The first systematic work dealing with the organic chemistry of silicon was carried out at University College, Nottingham, in the laboratory and under the direction of Professor F. S. Kipping. The basic research programme which he initiated, and which was to extend over a period of some forty years, started with a study of the stereoisomerism of organosilicon compounds. However, continual extension of this aim resulted in the foundation of what was virtually a new branch of chemistry. Kipping's work did establish the potential asymmetry of a silicon atom to which different organic groups are attached, and he was able to prepare optically active organic compounds of silicon. Continually, however, his preparative work was hindered by the formation of oily, rubbery, and resinous materials, which he was unable to crystallise. Recognising many such compounds to be condensation products containing Si-O linkages, Kipping gave to them the generic term *silicones*. The word has been retained, and is at present applied to organosilicon polymers or, more correctly, organopolysiloxanes containing the characteristic Si-O and Si-C linkages. The silicon "esters", containing only Si-O bonds, are not normally regarded as silicones.

Whilst Kipping was unable to foresee any industrial significance for his work and, indeed, reported to the Royal Society in 1937 that: "the prospect of any immediate and important advance in this section of chemistry does not seem to be very hopeful", chemists of the Corning Glass Co. of America were, in the late thirties, beginning to examine the by-products to which

Kipping had ascribed little importance. Laboratory examination of these products convinced those workers that silicones could play an important rôle in industrial life, and in 1943 the Dow Corning Corporation was formed, the specialised manufacturing knowledge of the Dow Chemical Company having been sought a year earlier. Thanks to the genius and the industry of American applied research teams, little time was allowed to elapse before silicones were being produced, and sold, in bulk.

Silicone production may be regarded, broadly, as a two-stage process. Firstly there is the production, separation, and purification of the intermediate organochlorosilanes, followed by hydrolysis and condensation to yield polymers. The chlorosilanes are produced by two main methods, the classical Grignard reaction, modified by Kipping for organosilicon synthesis, and the so-called "direct" method—



The chlorosilanes produced in the first step of the reaction form an exceedingly complex mixture, and must be separated and purified by a highly efficient fractionating column, before they are subjected to hydrolysis and condensation. This distillation process is made more complicated by the relatively close boiling points of these chlorosilanes and by their irritant, corrosive, and inflammable nature. Additionally, water must be rigorously excluded from all systems, other than

the actual hydrolysis plant, containing chlorosilanes, which may otherwise be spontaneously polymerised to the detriment of the processing plant. The general formulae R_3SiCl , R_2SiCl_2 , and $RSiCl_3$ represent the three categories of chlorosilanes of major interest in the production of silicones, since the tetrasubstituted silanes R_4Si cannot be hydrolysed to form polymers and hence are not useful in this context. On hydrolysis, R_2SiCl_2 alone can only dimerise, and this type of intermediate finds its uses as an end-blocker or to form the dimer, which in the case of the methyl series is a volatile liquid. From the intermediate R_2SiCl_2 are derived the silicone fluids $R_3Si-O-(SiR_2-O)_n-SiR_3$, R_2SiCl_2 being used in the hydrolysis mixture to help stop the polymerisation at a desired fluid viscosity. The value of n may vary within very wide limits in fluids ranging in viscosity from 0.65 to $>10^6$ centistokes.

Linear polymers are used also in the production of silicone rubbers, compounds, and greases. Silicone resins are derived from the trifunctional intermediates, which, on hydrolysis and condensation, yield cross-linked networks.

The properties associated with the linear dimethylpolysiloxanes, more commonly known as dimethyl silicone fluids, typify those of all the silicone products of interest to the textile industry. They include—

- (a) Flat viscosity-temperature curves
- (b) Outstanding oxidation resistance
- (c) Inertness to many chemicals
- (d) A high degree of water-repellency.

The last named property is, of course, that best known in the textile industry. However, in order to develop to the full a permanently water-repellent surface, the dimethyl silicone fluids require a curing schedule of 2-3 hr. at 250°C ., which immediately limits their application to substrates, such as glass and ceramics, which will suffer no deterioration on exposure to that temperature. A marked characteristic of a glass surface so treated is that the silicone film, formed on the glass surface, causes appreciable lowering of the coefficient of friction of the glass, is resistant to solvent attack, and induces water to coalesce into small droplets of high angle of contact.

That these properties are most desirable in a water-repellent finish for textile fabrics was soon appreciated, the lowering of coefficient of friction being associated with the soft, silk-like handle which is a characteristic of many silicone-treated materials. It remained, therefore, so to modify the basic silicone that the curing temperature could be reduced to such a value that no deterioration of a textile fibre would take place. There are a number of methods whereby this can be achieved. Thus it is possible to replace the methyl groups in a methyl silicone by alkoxy groups. Those groups which are water-sensitive and whose further polymerisation can be achieved by acid or alkali catalysis, however, necessitate the application of such a silicone from solvent (other than water) solution. Again, it is possible to use unsaturated

groups which, by oxidative or addition polymerisation in the presence of suitable catalysts, could be made to form a tightly bonded film sheathing a textile fibre. The replacement of certain methyl groups by hydrogen, and the use of a cyclic silicone whose ring could easily be opened by heat and catalysis, will both yield silicone polymers which can be made to polymerise further and yield water-repellent films. In the first instance, the use of a methyl polysiloxane which contained a certain proportion of hydrogen atoms was adopted as the most suitable silicone product for application to textiles in order to render the latter water-repellent. Curable within a few minutes at 150°C ., such a product imparts a high degree of water-repellency, which is resistant to the action of dry-cleaning solvents, but which, unfortunately, suffers from a major defect, in that it accentuates marking-off on fabrics such as acetate rayon. Accordingly, further modification was necessary before a product which did not suffer from this disadvantage became available commercially. The silicone which embodied this final modification soon became available in the form of a solution in methylene chloride or hydrocarbon solvent in which the active silicone content was 65%. Such a solution may be used diluted with a suitable solvent, provided that arrangements have been made for the recovery of the solvent, or the solution may be emulsified, using recommended emulsifying agents, when required.

Emulsifying agents are chosen which are destroyed by heat, or which decompose, leaving a hydrophobic residue on the cloth, so that a major advantage of the silicone treatment, namely that a fabric which has been silicone-proofed requires no after-scour, is not lost. The cloth is padded through a bath of silicone content such that, after mangling, 1.5-4% of silicone is left on the fabric, which is then stenter-dried and baked for 5-10 min. at 150°C . A requirement for the curing oven is that it should have adequate fresh-air circulation so as to ensure complete oxidative polymerisation of the silicone. The curing temperature may be reduced slightly by the use of organometallic catalysts when certain emulsifying agents are used. Pick-up and curing schedule will depend upon the structure and the weight of the fabric. The general nature of the silicone film so formed has been fairly conclusively established. Aided by the low surface tension of the silicone, each fibre is covered by a polymer which, owing to further oxidative polymerisation, yields a tightly bonded film.

It has not been shown that chemical interaction between the silicone and the substrate takes place, although this possibility cannot be overlooked. In any case, the film formed is resistant to solvent attack, increases the abrasion resistance, and usually imparts a desirable soft handle as well as enhanced draping qualities to a fabric so treated. An interesting side-effect, especially on cellulose acetates, is that ironing temperatures can be increased by 10°C . or more without fusion of the material. Of even greater significance is the resistance to water-borne stains. Originally the silicones were applied to artificial fibres such as

nylon, acetate rayon, and Terylene for which hitherto no satisfactory water-repellent had been available. In addition to fashion rain-wear and sports-wear, these desirable side-effects have given rise to a wider application of silicones where the quantity used is less than that required to develop full water-repellent characteristics.

The use of the silicone in such a form leaves something to be desired, however, since it is not a very simple matter to make a good emulsion unless special equipment, such as a colloid mill or a homogeniser, is available. Unless an emulsion of small particle size and good stability is prepared, moreover, unsightly oil spotting of the fabric can arise. Again, emulsifying agents have to be chosen such that, if desired, other finishing aids can be employed in the same bath. For example, on certain warp-faced acetate rayon materials, any tendency to slippage is accentuated by the lubricating action of the silicone film. Since anti-slip agents incorporated with emulsions of the solvent solution of silicones did not always give satisfactory performance, laboratory work was directed towards producing an emulsion, which could be marketed, having a storage life of a few months and completely compatible with a wide variety of finishing aids. Such a silicone emulsion is now available and has widened considerably the scope of water-repellent applications of silicones to textiles.

Requiring only the addition of a catalyst emulsion and dilution with water to the required silicone content, the stable emulsion, containing 40% of silicones, enables finishers who possess neither emulsifying equipment nor solvent recovery plant to make use of the silicone water-repellents. There is, however, a much greater significance in the production of such an emulsion, for the latter is so formulated that a wide variety of finishing aids are compatible, including stiffening and anti-slip agents and crease-resisting resins of the urea- and melamine-formaldehyde types. Economically desirable, a one-bath crease-resisting and silicone water-repellent finish has thus become practicable, and may be applied to the cellulosic fibres, viscose rayon and cotton, as well as to mixtures of these with other fibres. Fabrics so treated are softened by the silicone, which is usually applied so as to give a 3-4% pick-up on the cloth. Some attention must be paid to the preparation of the impregnating bath, and usually the correct order of addition involves adding the resin catalyst to the diluted resin. The mixed silicone emulsion and its catalyst are then added, and final dilution is completed. Compatibility with most of the common catalysts used to polymerise urea- and melamine-formaldehyde precondensates is a feature of the silicone emulsion, but in cases of doubt this compatibility can easily be ascertained by laboratory tests.

The application of a silicone together with a crease-resisting resin on rayons and cottons involves the formation of an external silicone sheath on each fibre, the resin precondensate entering the fibre and polymerising there in the usual fashion. However, if the best results are to be obtained, careful control of the process is

desirable. The water-repellency of the finished material may be impaired by the presence of large amounts of surface resin, which interfere with the adequate coverage of the fibres by the silicone. Thus it is necessary to ensure complete wetting out and careful control of drying and baking schedules. Of equal importance is the possibility of volatile amines being formed as a result of improper application of the one-bath finish. For, whilst the application of a crease-resisting resin alone usually entails a cure of about 2-3 min. at 140°C., it is essential that the silicone be fully cured, which necessitates heating for at least 5 min. at 150°C. It may thus become necessary in certain cases slightly to modify the existing process so as to minimise the dangers of fishy odours arising. Whilst no additional after-scour is essential because the silicone has been used, the normal after-scour to remove free formaldehyde and low molecular weight material, and to neutralise residual acidity, is not thereby eliminated. Thorough rinsing of a fabric treated by the one-bath process is necessary if the water-repellent character of the silicone treatment is not to be masked by residual soap used in such scours. Similarly, if a fabric already made crease-resistant is to be finished with a silicone water-repellent treatment, soap or wetting agents left on the material may interfere with the water-repellency of the latter. An efficient scour is also necessary, for the reasons given above, before the silicone is applied. Besides achieving a high degree of water-repellency, the fabric will be found to have the soft handle associated with the normal silicone proof. A modification of the silicone emulsion to yield a crisper finish also has been carried out, and this emulsion type is now available for evaluation.

Perhaps of especial significance in Scotland is the development work being carried out on woollen fabrics and on heavy textiles. Work has been directed, in each case, towards the production of silicone products which require lower temperatures for polymerisation of the silicone than the 150°C. used in the present emulsions. Some success has attended these efforts, and emulsions capable of being cured for longer periods at 120-130°C. are at present being evaluated as water-repellent and shrink-resistant treatments for wool. A silicone in solvent solution which air-cures in a few hours is being evaluated as a proofing agent for heavy textiles and also as a commercial reproofing product available to dry cleaners. The application of silicones in such fields is still in its infancy, although first reports are encouraging.

Silicone rubbers, too, have been developed for rendering textiles waterproof. At present they suffer from two major disadvantages—poor adhesion compared with organic rubbers and plastics, and high price. However, when the former problem has been successfully overcome, the latter may be discounted, since, in spite of their initial high cost, such rubber coatings will find specialised uses where the special characteristics of silicones will be taken advantage of.

All that has been said so far has dealt with the finishing of textiles using silicones, but, as indicated

earlier, many other uses for silicones abound in textile processes and machinery. Silicones exhibit marked surface activity and find extensive application as anti-foaming materials. Fairly high cost is more than offset by the small amounts necessary to control foaming media. It need hardly be emphasised that equipment which cannot be used to maximum capacity is costly equipment. Thus, to permit dyeing, scouring, and sizing baths, in which foaming arises, to be used to their fullest capacity, a small quantity of silicone anti-foaming compound may be employed. A few parts per million of such a compound is often efficient, for example, in certain printing pastes and in soap-perborate scouring baths.

Such anti-foaming compounds, which are inert and non-toxic, are available as grease-like materials, normally applied from solution in a suitable solvent, or from emulsion. Recently interest has been aroused in America in the use of an aerosol packaging of such materials, and where foam is localised or intermittent, a silicone anti-foaming agent available in such a form will probably find acceptance.

A recent development of a silicone release agent specifically designed to meet the needs of the textile industry is a solvent solution containing 25% of siloxane, which when applied to drying cans eliminates plucking of warp-sized yarns as well as build-up of the sizing material on the can itself. This release agent is applied by spraying or brushing, and concentrations of 5-10% and 10-15% respectively of silicone have been found to provide effective release surfaces. The release agent is used in conjunction with a catalyst consisting of an organolead salt which causes the silicone to cure overnight at room temperature or after 30-120 min. at 75-90°C. Such a release surface is semi-permanent, and operational times of 6-7 weeks per application have been obtained. Removal of the film may be achieved by rubbing with a solvent-soaked rag or brush.

Silicones, too, are playing an important part in machinery maintenance in the textile mill. Silicone fluids of themselves are deficient in lubricating power under any except light loads, by virtue of their inert nature. Thus under conditions of boundary lubrication there occurs no migration of surface-active material to adjacent surfaces. Work in this respect has been devoted to establishing which combinations of metals may successfully be lubricated by silicone fluids so as to overcome metal-metal friction. Additives may also be used, and, whilst these may tend slightly to reduce the outstanding characteristics of the silicones, the silicone greases are produced by compounding silicone fluids stable to high and low temperatures, with fillers such as metallic soaps or carbon black.

Those greases which retain their consistency over a wide range of temperatures and under moist conditions may be used under medium or light loads to replace conventional organic greases. Thus, in one textile mill, a silicone grease used to lubricate ball bearings, in a drying can, operating at 400 r.p.m. around saturated steam journals,

was found to have 45 times the life of the best organic lubricant used. A problem of a somewhat different type is encountered in the lubrication of valves and stop-cocks used in pipe-lines conveying hot or corrosive chemicals or steam. Here, lubrication is of secondary importance compared with the ability to withstand chemical and physical degradation. For such purposes, silicone compounds filled with silica in order to attain a grease-like consistency are available. These compounds withstand heat, moisture, and attack by a large variety of chemicals both in the liquid and in the gaseous state. The inert nature of such compounds has widened their uses to include lubrication and protection of rubber and plastics gaskets and packings.

Mention may be made here of the use of silicone sewing thread lubricants which may be applied neat, from aqueous emulsion or from solution in an organic solvent. Applied to sewing threads in low concentration, such lubricants reduce needle temperature with subsequent needle hole fusion when thermoplastic synthetic fibres are being sewn.

The stability to high and low temperatures and the moisture-resistant characteristics of the silicone fluids and greases are also typical properties of the silicone resins and rubbers. Those properties taken in conjunction with the good electrical properties and resistance to corrosion of these silicone products fit them for use as electrical insulants. Motors being operated in a textile mill may be especially prone to failure, and even to causing fires, owing to proximity to processing equipment used at high temperatures. Perhaps of even greater importance in this respect is failure of insulation by tracking or interference with cooling of the windings by dust or fibre particles. Similarly, a common cause of motor failures in textile mills is the high humidity which often prevails. Class H insulation, well established both in this country and in America, which utilises silicone resins, varnishes, and rubbers, permits continuous operating temperatures of 180°C. This temperature limit is 50°C. higher than that allowed with Class B insulation, the most heat-stable insulation prior to the advent of silicones. Thus one can achieve a greater power: weight ratio or machines can withstand heavy overloads if silicone insulation is used. Similarly, breakdowns due to overheating or moisture are remarkably reduced when motors so insulated are installed.

The foregoing is but a brief cross-section of the applications of the available silicones which are of immediate interest to textile people in general, but, of course, new developments in the textile industry as well as in the silicone field must ensure that silicones will play an increasing part in the processing and finishing of textiles in years to come.

TECHNICAL SERVICE DEPARTMENT

MIDLAND SILICONES LTD.

BARRY

GLAMORGAN

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The Printing of Wool, including the Mélange or Vigoureux Printing of Other Staple Fibres

J. A. POTTER

Meetings of the Huddersfield Section held at the Co-operative Society Café, Huddersfield, on 21st December 1954, Mr. J. Calvert in the chair; and of the Leeds Junior Branch held at the University, Leeds 2, on 8th February 1955, Dr. C. B. Stevens in the chair

The principles of textile printing are outlined, as a background to a discussion of the preparation and the composition of printing pastes for application to wool cloth and the selection of dyes for this purpose. The technique of printing wool in the manner originated by Vigoureux is also described, and the selection of dyes and the composition of the printing pastes are discussed. The extension of this technique to recently developed synthetic fibres and to fibre mixtures is considered.

HISTORICAL

Textile printing is assumed to have originated in the East, where Chinese and Indians practised hand printing with wooden blocks more than two thousand years ago. Cloth from the tombs of the ancient Egyptians shows that they, too, were able to print textiles.

Modern textile printing in Europe can be traced from India, through the Near East, reaching Germany, France, and England by the latter part of the seventeenth century.

Printed styles can be produced by one of four methods—

- (1) Hand-block technique
- (2) Surface printing machine
- (3) Roller or machine printing
- (4) Screen printing.

As this paper is concerned mainly with the printing of wool, the application of each of these methods to wool cloth will be briefly described. Each method has certain advantages and certain disadvantages, and no one technique can be employed for all types of printing.

HAND-BLOCK PRINTING

An engraved hard-wood (lime, plane, sycamore) or metal (copper or brass) block is used. Several thin pieces of wood or metal are screwed together to make the block, which can be of any size and 2-3 in. thick. The design is sketched on the plain surface of the block, and then engraved, leaving all lines in relief. Not all the pattern is engraved on one block, but just the part to be printed in one colour, the number of blocks required depending on the number of colours to be printed.

A solid table covered entirely with a felt pad and then American cloth is required, on which is laid the cloth to be printed.

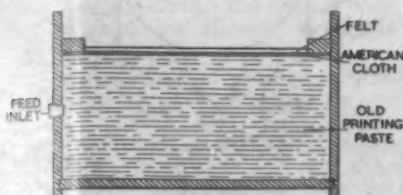


FIG. 1.—Sieve

The colour is transferred to the block by a piece of apparatus, known as a sieve (Fig. 1), consisting of a wooden box, covered at the top with a layer of American cloth and a layer of felt. This box is filled with old printing paste in order to give support to the felt, which carries the actual printing paste.

The colour is spread evenly on the upper surface of the felt with a brush. The block is then placed on the printing paste, which adheres to the relief surface and is thus transferred to the cloth to be printed. On each block there are four pins, one at each corner, which mark the cloth; each block must be inserted with its pins in the marks left by the preceding blocks, thus ensuring registration of the pattern, which is built up colour by colour.

When completely printed, the cloth is dried and steamed to fix the dyes, and finally washed and soaped to remove the thickening and unfixed dye.

The advantages of block printing are that it gives bold effects, excellent penetration, and full rich colours. Disadvantages are that fine lines and sharp definitions cannot be obtained and—a more important point—production is slow and expensive.

This method is mainly of historical interest, there being now very few printers who use block printing. One of the main uses is the production of imitation leopard-skin effects on pile fabrics.

ROTARY BLOCK OR SURFACE PRINTING

This is a far more rapid process, and the pattern is raised in relief on wooden rollers instead of on wooden blocks. These printing rollers revolve between flannel-covered copper cylinders, the lower of which rotate in troughs containing the printing paste (Fig. 2). The cloth to be printed passes between the printing rollers and the upper covered copper cylinders, and picks up the colour from the regions which are in relief.

In most machines there are a number of synchronised rollers each printing one colour, so that a complete multicolour pattern can be built up.

The advantages of block printing mentioned above are to some extent achieved, but with more rapid production. Disadvantages remain, such as the impossibility of obtaining fine lines.

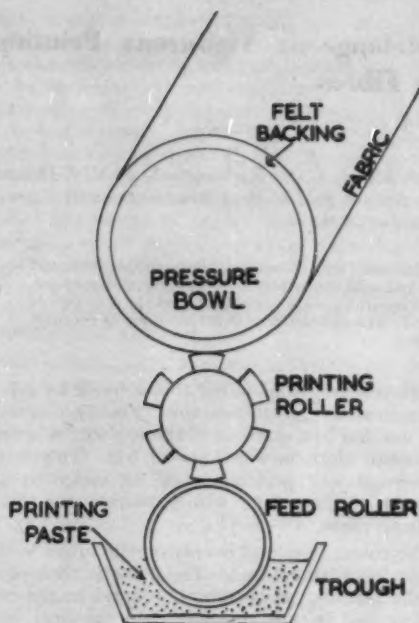


FIG. 2

MACHINE OR ROLLER (INTAGLIO) PRINTING

This is by far the most widely used method of printing, since, after the initial cost of engraving copper rollers, the labour costs are very low indeed compared with other methods.

The roller printing machine can be best understood by reference to Fig. 3 and 4, which show diagrammatically sections of a single-colour machine and a six-colour machine respectively. A hollow copper roller engraved with the pattern to be printed is carried by a steel shaft or mandrel, and revolves in contact with the pressure bowl above and the colour-furnishing roller below, the latter being partly immersed in the colour box.

A sharp-edged steel blade known as the *cleaning doctor* rests upon the engraved roller, and serves to scrape superfluous colour from the smooth parts of the roller, leaving the paste only in the engraving. A second doctor blade, made of brass, is arranged as shown against the engraved copper roller, and serves to remove the lint and nap which detach themselves from the cloth and stick upon the moist surface of the printing roller. This doctor is known as the *lint doctor*.

The central bowl is wrapped with several thicknesses of special cotton fabric called *lapping*. An endless woollen blanket circulates round the pressure bowl, and gives additional springiness to the surface. The *backgreys* or *back-cloth* is interposed between the blanket and the cloth to be printed for the purpose of preventing the former from being soiled by colour which penetrates through the latter. A rubberised blanket can be used in place of the backgreys.

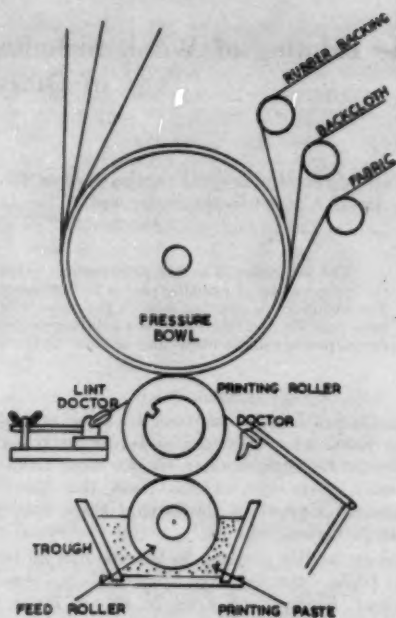


FIG. 3

With multicolour machines the same principle is used, each printing roller being in contact with the same pressure bowl.

After printing, the cloth is dried over steam-heated drying cylinders and is then steamed to effect fixation or development.

From a single machine the output per day can be 18,000 yd. with a single-colour machine and 8000-9000 yd. with a multicolour machine. This method is, therefore, the most widely used in

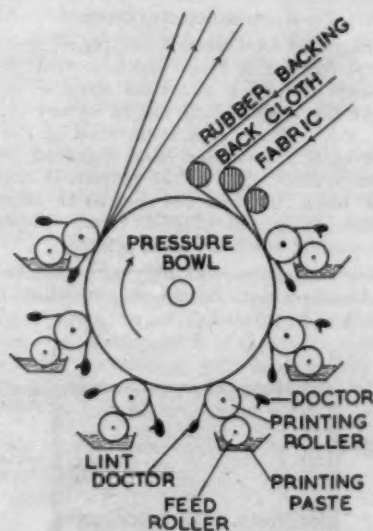


FIG. 4

printing all classes of textiles. The disadvantages lie in the high cost of roller engraving, and also capital machine cost, which preclude use of this form of printing where only limited production of a style is required.

SCREEN PRINTING

This method of printing has been in use for about forty years, although it is really a development of an extremely old form known as *stencil printing*. One big disadvantage of stencilling is the necessity of "ties" to connect the unsecured portions of the stencil, as shown in Fig. 5. If the non-porous laminar material, from which the stencil is cut, is replaced by a piece of fine silk gauze stretched across a frame and portions are painted out to obstruct the passage of printing paste, a screen is produced. In practice, fine bolting silk is stretched tightly over and fixed to a rectangular wooden frame. A separate screen is required for each colour in a multicoloured pattern.



FIG. 5

The simplest way to produce a screen is to trace the design on the silk and paint out the portions not required to be printed, but usually it is produced photographically. This is done by coating the surface of the bolting silk with a gelatin solution of ammonium dichromate known as *dope*, which is then exposed below a negative to an arc lamp, which renders the exposed gelatin insoluble. The unexposed dope is removed by washing in warm water. The insoluble gelatin is protected by lacquering one side of the screen completely and removing the lacquer from the clear areas with a solvent, e.g. methylated spirits, applied from the unlacquered side.

Screen printing is carried out on long tables covered with felt and protected with water-repellent American cloth. The cloth to be printed is laid along the table and attached to it with an adhesive. The print paste is spread evenly over the screen by means of a rubber squeegee, which forces it through the gauze on to the cloth at all points which are not blocked out. This is successively repeated down the full length of the fabric, stops on a guide rail being used to ensure registration of the pattern. The cloth is dried on the table, removed, and steamed as with other methods of printing.

The main disadvantage of screen printing is the high labour cost, since two operatives are usually required to carry out the printing operation. Within recent years mechanisation of the process has been developed to reduce labour costs, although screen printing is still generally done by the manual method.

Screens are very cheap to make in comparison with the cost of engraving copper rollers, so that this form of printing is ideal for designs of only a restricted output (e.g. head scarves on topical subjects).

THE PRINTING OF WOOL PIECES

With wool, apart from normal preparation processes such as scouring and, if necessary, bleaching, chlorination is always required in order to assist wetting and dye absorption.

Wool may be printed with most types of dyes, with the exception of those requiring strong caustic alkali in their application. All types of dyes used in wool dyeing, therefore, can be used in wool printing. Direct, basic, and leuco vat esters also may be used.

Dyes as such or as solutions cannot be used for printing. It is necessary to make up a paste, the viscosity of which will allow definite outlines to be obtained. This is done by the use of a thickening agent such as British gum, gum tragacanth, or a combination of the two. Other chemicals added to printing pastes are wetting-out agents, acids, and products to assist fixation during steaming. In wool printing urea is not always used, since it tends to harshen the wool. With chrome dyes, a chromium salt must, of course, be added. Typical recipes for acid and Benzyl Fast (Cloth Fast), Chrome Fast, and metal-complex dyes are given in Table I.

TABLE I
Printing of Wool Cloth

| | Acid and Benzyl Fast | Neolan | Chrome Fast | Cibalan |
|-------------------|----------------------------|--------|----------------|---------|
| Dye, g. ... | 30 | 30 | 30 | 30 |
| Urea, g. ... | — | — | 100 | 100 |
| Solvent, c.c. | 50 | 50 | — | — |
| Glycerol, g. | 40 | 40 | 40 | 40 |
| Water, c.c. ... | 110 | 110 | 110 | 110 |
| Thickening, g. | 600 | 600 | 600 | 600 |
| Acid, c.c. ... | 70 | 70 | 70 | — |
| Chromium salt, g. | — | 60 | 100 | — |

Leuco vat esters are used in wool printing, but the technique is somewhat more complicated, and will not be dealt with further here.

After printing it is necessary to steam. This is usually effected in a strong cylindrical chamber, arranged so that steam enters under a false bottom and also so that no condensation occurs on the wool cloth. Moist steam is essential, and it is usual to steam at 100°C., i.e. virtually at atmospheric pressure. Alternatively, a Mather & Platt continuous steamer or ager may be used. It is essential not to steam under pressure to avoid yellowing and damaging the wool.

After steaming, the wool cloth is washed to remove the gum and surplus colour, and drying and finishing are carried out in the normal manner.

MÉLANGE OR VIGOREUX PRINTING

Mélange printing is in effect the printing of slubbing or sliver, for subsequent processing to yield a "mixture" effect.

The definition of *mélange yarn* adopted by the Textile Terms and Definitions Committee of the Textile Institute is "a yarn produced from coloured printed tops or slivers, being distinguishable from a mixture yarn in that the fibres have more than one colour upon them". Mixture yarns, of course, are produced simply by blending dyed and undyed slubbing, and this is the easiest and simplest way of producing a mixture effect. However, *mélange* yarns, since each fibre has more than one colour upon it, give a very different appearance to the finished cloth. They produce a uniformity of shade quite unobtainable by the usual method of mixing dyed and undyed fibres. The latter, particularly in black-and-white mixtures, give a somewhat streaky appearance, which cannot be overcome even when the utmost care is exercised, and no matter how many times they pass through the gill box. It has been said, by one authority, that the reason for this is that whole fibres of coloured and white tend to ride on the surface of the cloth. The effect of *mélange* on the finished yarn is akin to that which would be obtained by blending together very short fibres.

This process is referred to also as *Vigoureux printing*, after the name of the inventor, who took out patents¹ as far back as 1863, although there appears to be no recorded development of the process up to the early 1890s. The patent rights were granted to the Industrielle Vigoureux of St. Denis in 1863, but extension of the work was very limited because the patentee reserved to himself the exclusive use of his rights during the entire period of the grant². As soon as this had expired, however, an important firm in Roubaix took up the novel invention, and, in due course, a wide variety and a large number of other companies followed suit. Without doubt, this expansion of interest must have taken place during the 1880s, because in 1892 Ostersetzer³ wrote that: "the process by which slubbing is printed on roller-type machines for the eventual production of mixture yarns has been operated for the past few years in both France and England, and was quite recently introduced into Germany".

Since that date, the Continental home of Vigoureux printing has remained in France, but even there the offspring in the Roubaix area steadily eclipsed the parent in St. Denis. Unfortunately, there is no published biography of Vigoureux himself, and his work appears to have developed with no change in the principles and only minor variations in the practical details.

The photomicrographs in Fig. 6 and 7 show the different appearances of fibres from a *mélange*-printed mixture yarn and from a mixture yarn obtained by blending separately dyed fibres respectively. In Fig. 6 it can be clearly seen that a single fibre has coloured and uncoloured regions; whereas in Fig. 7 individual fibres exhibit no such effect, and are either uncoloured or completely coloured.

The general principles of the printing of slubbing are the same as for wool cloth, except that chlorination is inadvisable, since the mechanical

properties of the slubbing must be such as to enable it to be spun and woven, and a chlorinated slubbing would not be suitable for the production of the highest-grade fabrics.

In this country it is usual to print on oil-combed tops, but too high an oil content may tend to yellow the wool, though this discoloration may be removed satisfactorily by suitable backwashing. On the Continent dry-combed tops in the main are processed. So far, it has not been possible to print more than one colour on the sliver. If several colours are required, the usual technique is to print the various colours on separate sliver, and blend them afterwards in the gill-boxes.

The *mélange* process gives a more intimate blend of coloured and uncoloured fibre. It is also claimed that a softer handle is obtained with the finished cloth, but the real reason for the present popularity of this type of printing is that effects are produced which cannot be obtained by blending dyed and undyed wool.

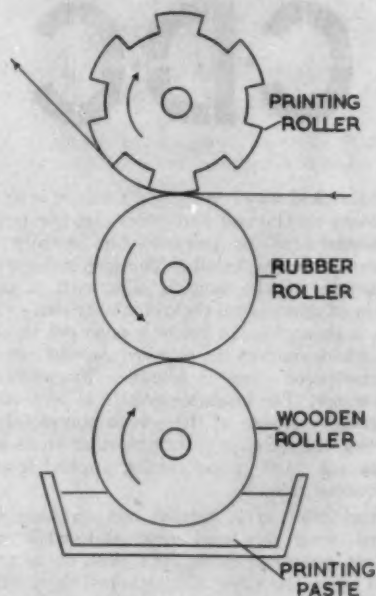


FIG. 8

The technique is very similar to the second method described above for cloth, viz. the rotary block. The usual type of printing unit in this country is shown in Fig. 8. A wooden roller, partly immersed in the printing paste in a wooden or stainless-steel trough, rotates against a felt-covered rubber roller. Printing paste is thus transferred to the latter roller. A brass doctor roller running against this removes surplus printing paste and ensures even distribution. The actual printing roller, which has on its surface elevated ribs inclined at an angle to the axis, is placed above and in contact with the felt-covered roller.



FIG. 6

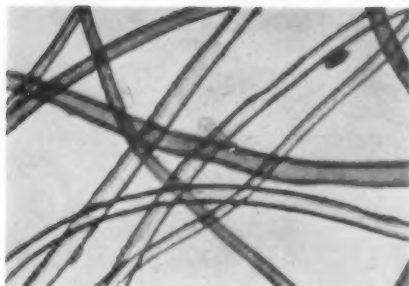


FIG. 7

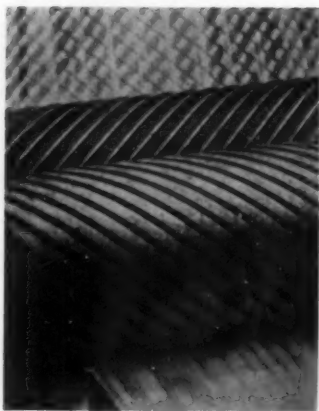


FIG. 10



FIG. 12



A thin web of the sliver to be printed passes between these two upper rollers. The pressure of the ribs of the printing roller transfers printing paste on to the fibre. In the spaces between the ribs, where no pressure is applied, no transfer of colour occurs. It is clear, therefore, that coverage of the sliver depends entirely on the ratio of rib to well. This can vary from 10 to 85%, and the ribs vary from $\frac{1}{4}$ to 1 in. in width.

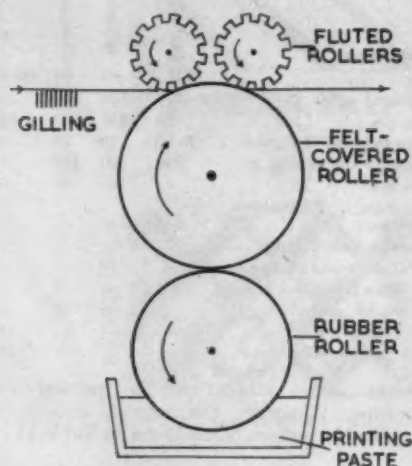


Fig. 9

Both Continental and present-day British machines are constructed with two printing rollers (Fig. 9), which operate simultaneously, thus producing a diamond pattern. If desired, of course, only one roller need be used, giving the line pattern on the sliver. As with the single-roller machine described first, the printing paste is picked up by a roller and transferred to a felt-covered roller, again running in immediate contact with the sliver. The two fluted bronze printing rollers with raised spirals are situated above the sliver, and again the printing paste is transferred to the wool at points where there is pressure. A pair of rollers are shown in Fig. 10, where the right-hand and left-hand spiral flutes can be seen.

The types of pattern produced by single and double rollers are shown in Fig. 11. The percentage of sliver printed again depends on the width of the spirals, and on the pitch of the flutes, although it is customary by makers to keep the pitch constant. Varying depths of colour may be obtained by suitable selection of rollers and by using them singly or together, the area of sliver printed thus being varied between 18 and 85%.

The sliver passes first through a gill box, then through the printing machine, and over an overhead cutting or plaiting device on to trays, ready for steaming.

The output of a printing machine per 8-hr. day can reach 880 lb., depending on the speed, working width of the gill box, and the machine used.

The techniques of making the printing pastes are very similar to those employed in printing wool cloth. However, it is more important to select dyes possessing good wet fastness properties, especially to alkaline milling, since the colouring is done prior to spinning, weaving, and finishing. Furthermore, good light fastness also is required, since mélange effects are used in the production of very good-quality suiting cloths. Therefore dyes are generally selected from the following classes—Acid Alizarine, acid milling, chrome, and metal-complex, e.g. Neolans and Cibalan. Typical printing paste recipes for these classes of dyes are given in Table II.

TABLE II
Vigoureux Printing of Wool

| | Chrome Fast | Benzyl Fast and Alizarine Fast | Neolan | Cibalan |
|--------------------------------------|----------------|---|--------|---------|
| Dye, g. ... | 30 | 30 | 30 | 30 |
| Water, c.c. ... | 490 | 490 | 490 | 490 |
| British gum, g. ... | 250 | 250 | 250 | 250 |
| Invadine BL conc. (20%), c.c. ... | 50 | 50 | 50 | 50 |
| Glycerol, g. ... | 40 | 40 | 40 | 40 |
| Sodium chlorate (25%), c.c. ... | 20 | 20 | 20 | 20 |
| Acid, c.c. ... | 20 | 90 | 25 | — |
| Chromium salt, g. ... | 90 | — | — | — |
| Oil of turpentine, c.c. ... | 10 | 10 | 10 | 10 |

It will be observed that very much less thickening agent is used than is normal for piece printing, in order to give good penetration of the sliver. In this form of printing good definition is of less importance. Whilst British gum is widely used as thickening agent, a Caragheen moss may be used, since this results in less adherence of the wool fibres and also may be more easily washed out afterwards. In mélange printing a wetting agent is necessary, again to assist penetration. As non-chlorinated wool is used, an addition of sodium chlorate is made to the printing paste to improve the yield. This also counteracts the reducing action of the wool and the thickening agent on many dyes. Glycerol is also added, mainly to prevent the printing paste drying before steaming, since this drying would make development difficult. Oil of turpentine is used as an anti-frothing agent.

By far the most common shade produced by mélange printing is grey, and for this a Chrome Black type is used, in combination with shading dyes. Dyes of the newer metal-complex types, e.g. Cibalan, are now widely used because of their excellent fastness to light and milling. These have the advantage over Chrome Fast dyes that their use simplifies backwashing. For certain brighter colours it is necessary to use dyes from the Neolan or Benzyl Fast ranges.

As in the printing of wool cloth, it is necessary to steam in order to fix the dye. A normal cottage steamer, with a hinged cast iron door and an inverted-V shaped roof to prevent condensation on the wool, can be used, and will take four truck-loads of slubbing.

On the Continent a wooden steam chest with a perforated double bottom is sometimes used, in which case a perforated steam coil emits steam at low pressure below the perforated false bottom. It is customary to pack the steamer as full as possible. In Italy the printed sliver is merely laid on canvas and then placed in the steamer.

With all types of steamer, the time of steaming depends on the quality of the wool and on the dyes used. With some dyes one hour may be sufficient, but it is usually better to steam for two one-hour periods with intermediate cooling. Two separate steamings, whilst giving improved colour value, tend to make the size of the plant prohibitive. Very humid steam is necessary to obtain the maximum yield from the dye, and steaming is carried out at atmospheric pressure.

As with piece printing, it is essential to remove the thickening and surplus colour from the wool after steaming. This is a difficult operation, particularly with chrome dyes, necessitating the use of a backwash unit of at least three bowls with heavy squeeze units between each. With Cibalan a three-bowl backwasher will suffice. In all backwashers powerful squeegee rollers are used between each bowl.

After steaming, the printed sliver is allowed to cool before backwashing. In the first and final bowls running water at 100–120°F. is used, whilst in the intermediate bowls a detergent at 100–120°F. is employed.

After backwashing, drying is carried out either by contact with steam-heated cylinders or in a hot-air dryer, overdrying being avoided because of the effect on the wool fibre.

After drying, the slivers go straight to a gill-box.

Terylene Polyester Fibre

Interest has recently been shown in the mélange printing of synthetic fibres, e.g. Terylene, and a typical recipe for this purpose is given in Table III.

TABLE III
Vigoureux Printing of Terylene with Cibac Dyes

| | | | | |
|-------------------------------|-----|-----|-----|----------|
| Dye | ... | ... | ... | 40 g. |
| Water | ... | ... | ... | 220 c.c. |
| Thickening | ... | ... | ... | 620 g. |
| Resist Salt C | ... | ... | ... | 10 g. |
| Invadine BL conc. (20% soln.) | ... | ... | ... | 30 c.c. |
| p-Hydroxydiphenyl (25% soln.) | ... | ... | ... | 80 g. |

When steaming under atmospheric conditions diffusion of dye is slow, and long steaming times are necessary to produce acceptable colour yield. The rate of dye diffusion may be increased by (a) steaming at higher pressure (and temperature) or (b) use of swelling agents ("carriers"). The effect of the latter is greatest at atmospheric pressure. At higher steaming pressures little advantage appears to be gained by incorporating swelling agents.

Viscose Rayon Staple

There has also been some interest in the mélange printing of viscose rayon staple. The main difficulty is caused by slippage of the sliver. A

normal type of backwasher cannot be used, though some recent types are said to be satisfactory. It is necessary to resort to the use of a "tape" backwasher, or alternatively to use a Hussong or Obermaier pack machine to wash the sliver after steaming.

TABLE IV
Vigoureux Printing of Viscose Rayon Staple

| Dye class | Coprantine | Cibagene | Cibanogene | Neosol |
|-----------------------------------|------------|----------|------------|--------|
| Dye, g. | 10–15 | 80 | 80 | 60–80 |
| Solubilising agent, g. | 150–200 | 130 | 110 | 180 |
| Water, c.c. | 200 | 250 | 180 | 430 |
| Caustic soda (66"Tw.), c.c. | 5–10 | 20 | 20 | — |
| Tragacanth thickening, g. | 550 | 500 | 480 | — |
| Water, c.c. | — | — | 40 | — |
| Ammonium dihydrogen phosphate, g. | 2–4 | — | — | — |
| Coprantine Salt TC, g. | 30 | — | — | — |
| Neutral chromate soln., c.c. | — | 20 | — | — |
| Cibanogene Developer S, c.c. | — | — | 80 | — |
| Fumexol AS, c.c. | — | — | 10 | — |
| Glucose (solid), g. | — | — | — | 80 |
| British gum powder, g. | — | — | — | 250 |

Viscose rayon staple can be printed with Coprantine, Cibagene, Cibanogene, and Neosol dyes, suitable recipes being given in Table IV.

Regenerated Protein Fibres

The mélange printing of fibres such as Fibrolane has also been considered. The most suitable dyes for this fibre are the normal types of wool dye, and selected Chrome Fast dyes have been found to give satisfactory fastness to milling.

Wool-Viscose Rayon Mixtures

In the case of wool-viscose rayon staple mixture sliver, when printed with mixtures of wool and cotton dyes, a typical recipe for which is given in Table V, the wool dye which falls on the viscose rayon is not fixed, and is removed during washing;

TABLE V
Vigoureux Printing of Wool-Viscose Rayon Staple Mixtures with Mixtures of Wool and Direct Cotton Dyes

| | | |
|-------------------------|-----|----------|
| Coprantine dye | ... | 40 g. |
| Benzyl Fast dye | ... | 40 g. |
| British gum | ... | 250 g. |
| Water | ... | 295 c.c. |
| Urea | ... | 150 g. |
| Lyoprint DA | ... | 50 c.c. |
| Invadine BL conc. (20%) | ... | 50 c.c. |
| Glycerol | ... | 40 g. |
| Sodium chlorate (25%) | ... | 20 c.c. |
| Ammonium oxalate | ... | 25 g. |
| Oil of turpentine | ... | 10 c.c. |
| Coprantine Salt TC | ... | 30 g. |

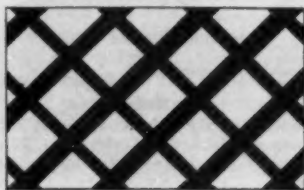
and similarly the direct cotton dye which stains the wool may not be completely fixed, and consequently it, too, is removed during washing. For this reason it may be more economical to print the wool and viscose rayon slivers separately, and mix them in the gilling operation. Alternatively, a



Roller No. 1—18%
(printed bars running to the left)



Roller No. 2—27%
(printed bars running to the right)



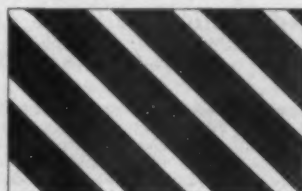
Rollers No. 1 and 2—40%



Roller No. 3—50%
(printed bars running to the right)



Rollers No. 1 and 3—60%



Roller No. 4—70%
(printed bars running to the left)



Rollers No. 2 and 4—78%



Rollers No. 3 and 4—85%

Direction of the wool after printing

FIG. 11—Percentages of the Surface Area printed²

TABLE VI

Vigoureux Printing of Wool-Viscose Rayon Staple Mixtures with Chrome and Direct Cotton Dyes separately

| CHROME FAST DYES | | |
|-------------------------------------|-----|-------------|
| Dye | ... | 30-80 g. |
| Water | ... | 490 c.c. |
| British gum | ... | 250 g. |
| Invadine BL conc. (20%) | ... | 50 c.c. |
| Glycerol | ... | 40 g. |
| Sodium chlorate (25%) | ... | 40 c.c. |
| Acid | ... | 20 c.c. |
| Chromium salt | ... | 90-140 c.c. |
| Oil of turpentine | ... | 10 c.c. |
| COPRANTINE DYES | | |
| Dye | ... | 15 g. |
| Urea | ... | 150 g. |
| Lyoprint G | ... | 50 c.c. |
| Water | ... | 373 c.c. |
| Caustic soda (66° Tw.) | ... | 10 c.c. |
| Neutral tragacanth thickening (6%) | ... | 350 g. |
| Ammonium dihydrogen phosphate (1:2) | ... | 12 c.c. |
| Coprantine Salt TC | ... | 40 g. |

mélange effect can be obtained by printing the blended sliver with dyes which have affinity for only one fibre, e.g. selected Chrome Fast dyes alone and selected direct cotton dyes alone. This method is more economical than printing the blend with mixtures of acid wool dye and direct cotton dye. Typical recipes are given in Table VI.

In the laboratory a hand-block technique can be used, as shown in Fig. 12, and this gives results reasonably comparable with those obtained on actual printing machines.

* * *

The author desires to express his thanks to the Directors of the Clayton Dyestuffs Co. Ltd. for permission to publish this paper and to his colleagues Messrs. J. C. Dunlop and T. Green for assistance with its preparation.

DYEHOUSE LABORATORIES

THE CLAYTON DYE STUFFS CO. LTD.

CLAYTON

MANCHESTER 11

(MS. received 1st June 1955)

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COMMUNICATIONS**The Adsorption of Some Organic Vapours by Wool and Nylon**

H. R. CHIPALKATTI, V. B. CHIPALKATTI, and C. H. GILES

Vapour-phase adsorption experiments have been made with a number of organic compounds on nylon and wool fibres. Preliminary tests showed that all hydroxylic compounds have very considerably greater affinity for the fibres than have other polar substances. Rate curves and/or isotherms have been determined for acetic acid, ethanol, methanol, phenol, and other compounds. It is shown that in absence of air and moisture the fibres are stable for long periods at temperatures as high as 150°C., so that adsorption tests can be made with substances of high boiling-point, e.g. β -naphthol. At these temperatures each successive adsorption-desorption cycle produces a rise in the adsorptive capacity of the fibre, and sometimes in the rate of diffusion of the adsorbate. The results appear to indicate a mechanism by which hydroxylic compounds are adsorbed as a monolayer on localised sites, probably by hydrogen bonding at the oxygen atoms of the amide or peptide groups.

This paper describes part of an exploratory investigation into the adsorption of some organic vapours by nylon and wool, made to discover whether the technique can be used—(a) to determine the importance of hydrogen bonding in adsorption of organic compounds by such fibres; (b) to study the properties of the fibres at high temperatures; (c) to study the physical state of dyes in the fibres; and (d) to study vapour-phase dyeing techniques at high temperatures. The results are summarised in Fig. 2-9 and Tables I-III.

It is known that in the liquid phase, and in the absence of water, non-solvents for the fibres can penetrate nylon or wool only if their molecules are smaller than that of *n*-butanol in the aliphatic series, or the anthracene molecule in the aromatic series^{1,2}; hydroxylic compounds are apparently adsorbed from solution largely through the medium

of OH...O hydrogen bonds of relatively high affinity, and non-hydroxylic compounds largely by weaker physical attraction¹. Less definite information is available concerning the mechanism of adsorption from the vapour phase. Most previous investigations reported upon vapour-phase adsorption by fibres have been concerned with water vapour³. The literature on this subject is extensive, but the interpretation of the results is still under discussion, and it is probable that different types of adsorptive mechanism operate at different degrees of saturation of the fibre⁷. It has frequently been suggested that water is adsorbed on localised sites, probably on the polar side-chains⁸ and/or on the peptide groups of

* Carlsen⁵ and McLaren and Bowen⁶ give useful reviews of the information on water vapour adsorption on textile fibres in general, and on proteins in particular, respectively. See also e.g. Melton and Hoover⁴, and Katchman and McLaren⁶ for reports of investigations upon water vapour adsorption by proteins and by amino acids and derivatives, including peptides, used as protein models.

wool^{9,10}; but Hailwood and Horrobin¹¹ obtained good correlation between published experimental data and a hypothesis of a three-phase condition of adsorbed water in a state of ideal solid solution in the fibre.

Little work has been reported upon the adsorption by fibres of gases other than water vapour. Amongst the principal investigations of this type, however, are those of Kanamaru and Chao¹² and Lauer¹³, who adsorbed a number of simple organic compounds, principally alcohols, in vapour form on cellulose and its derivatives; and King¹⁴, who adsorbed the vapours of methanol, ethanol, and *n*-propanol on wool. King¹⁴ and King and Cassie¹⁵ observed a sudden rise in the temperature of the fibre when the vapour was first introduced, but thereafter the temperature returned slowly to its initial level. The rise is caused by the evolution of heat in the reaction between the fibre and the adsorbate, and it was shown to have a significant effect in retarding the initial diffusion by reducing the equilibrium adsorption of the vapour. The temperature rise was greatest with water and least with ethanol, because the larger the adsorbate molecule the slower its rate of diffusion into the fibre, and therefore the more readily can the heat of adsorption be dissipated.

Larose¹⁶ has made measurements of the adsorption of gaseous hydrogen chloride on wool, to which reference is made below.

Experimental

APPARATUS AND TECHNIQUE

The McBain and Bakr¹⁷ spring balance was used, the apparatus being shown in Fig. 1. The reagent was contained in a thin sealed-glass capsule in the liquid-reservoir tube, and was released by dropping on to the capsule a stainless-steel ball-bearing, raised by a magnet. The vapour reservoir was connected to a Barr & Stroud two-stage high-vacuum lens-coating unit. The apparatus was evacuated to a pressure of 10^{-4} mm. of mercury (measured on a McLeod gauge). The spring* was made of Pyrex glass; this material gives very satisfactory results, provided that it is annealed under load at 200°C. for 8 hr. before use to ensure dimensional stability¹⁸. Pyrex glass springs are much more robust, easier to fabricate, and much less costly than the quartz springs usually employed for the purpose. The dimensions of a typical spring used in this work were—fibre diameter 0.008 in., coil diameter 0.75 in., 12 t.p.in., sensitivity 17.5 cm./g. with a 0.4-g. load. The extension was measured to 0.001 cm. by means of a cathetometer.

For the low-temperature experiments the whole apparatus was enclosed in an air thermostat constructed on the table of the vacuum unit. The high-temperature experiments were made in a modified apparatus in which the adsorption tube was immersed in a thermostatically controlled paraffin oil bath, the reagent reservoir being outside but in close proximity. Thus the reagent,

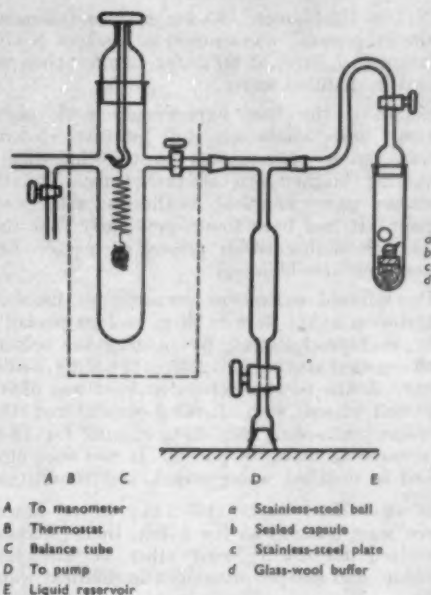


FIG. 1

if not very volatile, could be independently heated to a higher temperature than the bath, to accelerate the production of vapour. The bath was constructed of copper with a Pyrex plate glass window through which the spring was observed, the oil being circulated by a centrifugal pump through a copper coil brazed to the pump inlet and outlet orifices. In order to avoid condensation of liquid caused by the entering vapour cooling as it expanded into the adsorption tube, the vapour was first allowed to remain in the vapour reservoir for a short time before admission to the tube, the walls of which had been very slightly warmed by brief application of a luminous gas flame. Without these precautions, the spring registered an initial rapid increase of weight of the substrate on the impact of the vapour, followed by a slight decrease, before the normal steady increase commenced.

The degassing period was 4–5 hr. at the temperature to be used for adsorption. Adsorption results on different samples of nylon were reproducible; on wool small variations occurred from sample to sample, presumably owing to differences in fibre dimensions. Degassing of wool for an extended period (24 hr.), although causing no further decrease in weight, appeared to lead to somewhat lower adsorption rates, without affecting equilibrium values. In successive adsorption-desorption cycles with methanol vapour and wool the rate of desorption became progressively lower in each cycle. For the present isotherms fresh fibre samples were used for each determination.

The individual data given in this paper are in most cases the means of two or sometimes three replicates.

* Supplied by Messrs. Still & Cameron, Rugby Avenue, Wembley, Middlesex.

MATERIALS

NYLON (15-filament, 45-denier drawn yarn and undrawn monofil) was scoured in Lissapol N (ICI) solution (5 g./litre) at 60°C. for 15 min., then well rinsed in distilled water.

Some of the first experiments with phenol vapour were made on dull (titania-delustred) drawn nylon yarn, owing to the difficulty of obtaining bright yarn at that time. The two varieties gave identical isotherms with water vapour. It had been found previously that their respective isotherms for phenol adsorption from water were also identical.

Precipitated nylon was prepared by dissolving 1 g. drawn nylon fibre in 30 g. molten phenol at 60°C. and precipitating by pouring the solution with constant stirring into 2 litres of boiling distilled water. A fine powder separated, and was filtered off, well rinsed with distilled water, and then (Soxhlet-) extracted with 95% ethanol for 16 hr. to remove all traces of phenol. It was then again rinsed in distilled water, dried, and conditioned.

WOOL—Root ends (1.5–2 in.) of a Lincoln fleece were scoured as for nylon, then (Soxhlet-) extracted for 24 hr. with ether or methylene chloride, and steeped overnight in distilled water.

Before use, all fibres were air-conditioned for 24 hr., weighed, then oven-dried at 100–110°C. for 12–18 hr., and immediately introduced into the adsorption apparatus. The weights quoted here refer to the air-dry fibres.

ETHANOL and METHANOL were prepared anhydrous by refluxing for 12 hr. over quicklime, then distilling and refluxing over magnesium with addition of a little methyl iodide, and finally distilling into a receiver protected against atmospheric moisture. Some samples of methanol were

of commercial "specially dry" (B.D.H.) quality as used for Karl Fischer titrations.

ACETIC ACID (normally dried) was prepared by storing the analytical-quality material over anhydrous sodium sulphate. The intensively dried acid was prepared from this by refluxing for several hours with pure chromic anhydride (6 g./litre) and then fractionally distilling. The fraction boiling at 117.5–118.5°C. was collected in a flask fitted with a calcium chloride drying tube.

HYDROGEN CHLORIDE (dry) was prepared inside the adsorption apparatus by placing pure sodium chloride in the liquid-reservoir tube alongside pure sulphuric acid, contained in a thin-walled sealed-glass phial, and breaking the acid bulb.

ACETONE was dried over anhydrous cupric sulphate.

ANILINE was of B.P. quality, redistilled.

PYRIDINE was redistilled over sodium hydroxide.

Other reagents were of commercial analytical quality or were purified laboratory specimens.

SOLVENT DRYING OF WOOL

Investigations were made into the possibility of accelerating wool drying by preliminary treatments in anhydrous methanol to remove water, and then in dry benzene or dry ether to remove methanol; tests were then made of methanol vapour adsorption at 40°C. after the normal degassing procedure. Unfortunately, the results were disappointing.

Results and Discussions

COMPARATIVE ADSORPTION OF HYDROXYLIC AND NON-HYDROXYLIC COMPOUNDS

Several hydroxylic and non-hydroxylic compounds were examined to determine whether the

TABLE I
Preliminary Adsorption Tests

| Vapour | Fibre | Temp. (°C.) | Vapour Pressure Actual (mm. Hg) | Relative | Concn. of Adsorbate on Fibre C_F (m-moles/kg.) |
|-----------------------|-------|----------------|---------------------------------------|----------|--|
| POLAR, NON-HYDROXYLIC | | | | | |
| Acetone* ... | Nylon | 35 | 64 | 0.2 | Nil† |
| Acetone* ... | Wool | 22 | — | 1.0 | Nil† |
| Aniline‡ ... | Nylon | 70 | 11 | 1.0 | 260 |
| Azobenzene ... | Nylon | 100 | — | 1.0 | Nil (‡)† |
| Diethylamine ... | Nylon | 35 | 60 | — | Nil† |
| Dioxan ... | Nylon | 35 | — | 1.0 | 140 |
| Ethyl acetate ... | Nylon | 25 | 55 | 0.6 | 30 |
| Pyridine ... | Nylon | 15 | 10 | 1.0 | Nil† |
| Triethylamine ... | Nylon | 35 | — | 1.0 | 40 |
| Triethylamine ... | Wool | 35 | — | 1.0 | 55 |
| POLAR, HYDROXYLIC | | | | | |
| Acetic acid ... | Nylon | 28 | — | 1.0 | >1600 |
| Ethanol ... | Nylon | 26.5 | 40 | 0.75 | > 550 |
| Ethylene glycol‡ ... | Nylon | 100 | — | 1.0 | 380 |
| Methanol ... | Nylon | 26.5 | 94 | 0.7 | >1450 |
| Phenol ... | Nylon | 25 | — | 1.0 | >1150 |
| n-Propanol ... | Wool | 40 | 28 | 0.6 | > 510 |
| Water ... | Nylon | 26.5 | 16 | 0.75 | >1750 |

* See footnote* on p. 655.

† L.e. Adsorption below the limiting sensitivity of the method (ca. 10–20 m-moles/kg.).

‡ See also Table III (q, r) for details.

TABLE II
Adsorption-Desorption Cycle Rate Data

| <i>t</i> | <i>C_F</i> | <i>t</i> | <i>C_F</i> | <i>t</i> | <i>C_F</i> | <i>t</i> | <i>C_F</i> | <i>t</i> | <i>C_F</i> | <i>t</i> | <i>C_F</i> |
|----------|----------------------|----------|----------------------|----------|----------------------|----------|----------------------|----------|----------------------|----------|----------------------|
| (a) | (b) | (c) | (d) | (e) | (f) | (g) | (h) | (i) | (j) | (k) | (l) |
| 1 | 217 | 0 | 945 | 0 | 387 | 0 | 2401 | 1 | 827 | 0 | 3024 |
| 3 | 372 | 20 | 852 | 1 | 1285 | 1 | 2150 | 2 | 1087 | 1.5 | 2828 |
| 6 | 480 | 150 | 387 | 3 | 1595 | 3 | 2150 | 4 | 1545 | 15 | 2154 |
| 11 | 650 | | | 7 | 1905 | 30 | 1518 | 6 | 1884 | 40 | 1435 |
| 15 | 744 | | | 11 | 2401 | | | 8 | 2110 | | |
| 24 | 945 | | | | | | | 10 | 2328 | | |
| | | | | | | | | 18 | 2891 | | |
| | | | | | | | | 20 | 3024 | | |
| (g) | (h) | (i) | (j) | (k) | (l) | | | | | | |
| 0 | 1435 | 5 | 116 | 0 | 743 | 2 | 626 | 0 | 840 | 2 | 205 |
| 2 | 2502 | 12 | 232 | 12 | 255 | 12 | 812 | 10 | 750 | 16 | 513 |
| 6 | 3220 | 23 | 302 | 42 | 175 | 29 | 905 | 90 | 375 | 30 | 718 |
| 9 | 3556 | 35 | 395 | 62 | 0 | 65 | 1253 | 130 | 240 | 60 | 923 |
| 13 | 4003 | 75 | 534 | | | 92 | 1579 | 180 | 0 | 90 | 1060 |
| | | 150 | 743 | | | 112 | 1579 | | | 120 | 1197 |
| | | | | | | 130 | 1509 | | | 158 | 1368 |
| | | | | | | 144 | 1346 | | | 180 | 1436 |
| | | | | | | 167 | 840 | | | 210 | 1471 |
| | | | | | | | | | | 265 | 1573 |
| (m) | (n) | (o) | (p) | (q) | (r) | | | | | | |
| 0 | 1231 | 22 | 46 | 0 | 68 | 0 | 1460 | 0 | 2790 | 0 | 2077 |
| 3 | 1162 | 60 | 66 | 2 | 56 | 1 | 1310 | 1 | 1770 | 3 | 1863 |
| 23 | 1124 | 125 | 68 | 25 | 52 | 15 | 770 | 15 | 645 | 13 | 1530 |
| 80 | 1000 | | | 95 | 42 | 25 | 550 | 25 | 167 | 23 | 1391 |
| 140 | 1047 | | | | | 45 | 380 | 35 | 0 | 33 | 1275 |
| 180 | 992 | | | | | 90 | 100 | | | 60 | 1113 |
| | | | | | | 120 | 0 | | | | |

KEY
(Adsorption tests at saturated vapour pressure)

- a, b = β -Naphthol, drawn nylon, 120°C., first adsorption and desorption
 c, d = β -Naphthol, drawn nylon, 120°C., second adsorption and desorption
 e, f, g = β -Naphthol, precipitated nylon, 120°C., first adsorption, desorption, second adsorption
 h, i, j, k = Benzoic acid, precipitated nylon, 130°C., first adsorption, desorption; second adsorption, desorption
 l, m = Benzoic acid, wool, 130°C., adsorption, desorption
 n = Allazarin, wool, 130°C., adsorption
 o = Allazarin, wool, 130°C., desorption
 p = Methanol, drawn nylon, 26°C., desorption
 q = Water, drawn nylon, 26°C., desorption
 r = Intensively dried acetic acid, drawn nylon, 30°C., desorption.

former show any markedly higher affinity in the vapour phase for nylon and wool, as they do in adsorption from solution¹. The amount of each vapour adsorbed at or near equilibrium was determined in the exploratory experiments summarised in Table I. It is obvious that the hydroxylic compounds do show considerably greater affinity than the others, of which only two, aniline and dioxan, show any appreciable adsorption*. (The sample of aniline used was not intensively dried.)

ADSORPTION OF VAPOURS OF ETHANOL, METHANOL, AND WATER†

It was proposed to examine the adsorption of methanol vapour in particular, because it is one of the simplest and smallest hydrogen-bonding

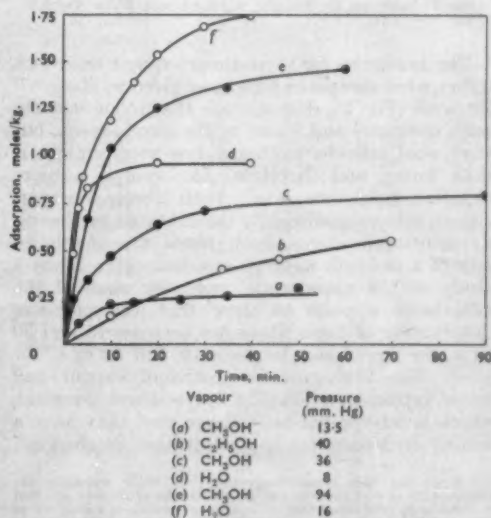
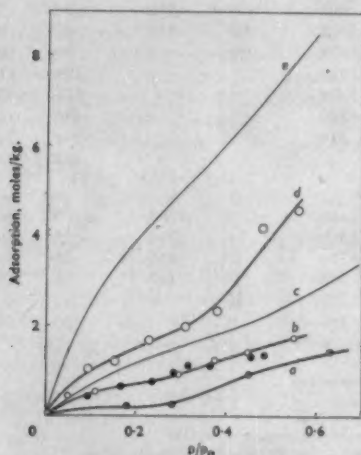


Fig. 2—Adsorption Rates of Ethanol, Methanol, and Water Vapours on Drawn Nylon Yarn at 26.5°C.

* Ordinary redistilled acetone was adsorbed to 80 m-moles/kg. on wool at 27°C. and saturated vapour pressure. King¹⁰ recorded adsorption figures for acetone on wool at 25°C. as high as those for propanol. His acetone, however, was dried over quicklime, which catalyses its decomposition to various hydroxylic compounds. In an experiment here, quicklime-dried acetone was adsorbed to over 350 m-moles/kg. on wool at 22°C. and saturated vapour pressure.

† After this paper had been prepared, a communication by Nichols and Speakman¹¹ appeared in which isotherms for water, methanol, ethanol, and n-propanol vapours on wool are shown. That for methanol is very similar to the present curve of Fig. 3 (d).

compounds. As a preliminary, its adsorption by nylon was examined in comparison with ethanol vapour and water vapour respectively, adsorption and desorption rate curves and isotherms being determined. The rates of both adsorption and desorption (Fig. 2; Table II*p*, *q*) increase in the order of decreasing molecular size, i.e. ethanol < methanol < water, as found by King^{14, 19} for wool. (In the present case the ethanol desorption rate was estimated only approximately.) The whole of the adsorbed methanol could be desorbed. (Speakman²¹ found that on wool it was impossible to remove the last 6% of methanol adsorbed from the pure liquid.)



(a) Ethanol on drawn nylon at 26°C.
(b) Water (○) and methanol (●) on drawn nylon at 26°C.
(c) Ethanol on wool (Australian merino) at 25°C. (King¹⁴)
(d) Methanol on wool (Lincoln) at 40°C.
(e) Water on wool (Australian merino) at 25°C. (King and Cassie¹⁹).

FIG. 3—Isotherms for Ethanol, Methanol, and Water Vapours

The isotherms for these three vapours on drawn nylon, when compared with those given by King^{14, 19} for wool (Fig. 3), demonstrate that nylon adsorbs both methanol and water to the same extent, but that wool adsorbs methanol less readily than it does water, and therefore that wool is a more selective molecular sieve. Both fibres, however, adsorb ethanol less readily than they do methanol; as mentioned above, both fibres are unable to adsorb a molecule as large as *n*-butanol*. Thus a study of the appropriate molecular sizes of the adsorbates appears to show that the pore-size distribution of these fibres lies between 15 and 30 sq.Å. for nylon and between 10 and 30 sq.Å. for wool. The isotherms for methanol vapour and water vapour on nylon (Fig. 3) are almost identical, which is interpreted as evidence that they have a similar hydrogen-bonding adsorption mechanism.

* There has been some disagreement^{19, 22, 23} regarding the penetrability of wool fibres by the larger molecules of alcohols, etc., but it seems to be established that the rate of penetration of a molecule as large as *n*-butanol under normal conditions is so low that the fibre may be considered impenetrable to it, and the normal pore-size may be estimated accordingly.

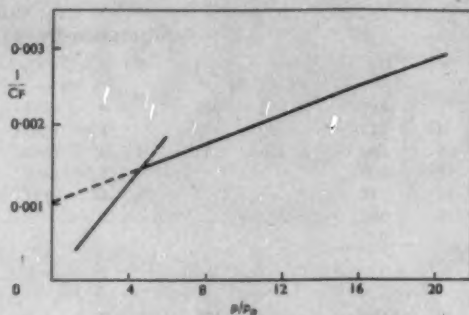


FIG. 4—Methanol and Water Vapour Adsorption on Drawn Nylon at 26°C.—Langmuir Plot (by Interpolation) of Data of Fig. 3

Fig. 4 shows Langmuir plots obtained by taking interpolations from the isotherms of Fig. 3. The maximum amount adsorbed is 1 mole/kg. for both water and methanol. If this represents attachment of one methanol molecule to one amide linkage*, then about 11.5% of the total number in nylon are accessible. The data of Fig. 3 are insufficient for a reliable determination of the value for ethanol on nylon, but it appears to be about one-third of that for methanol.

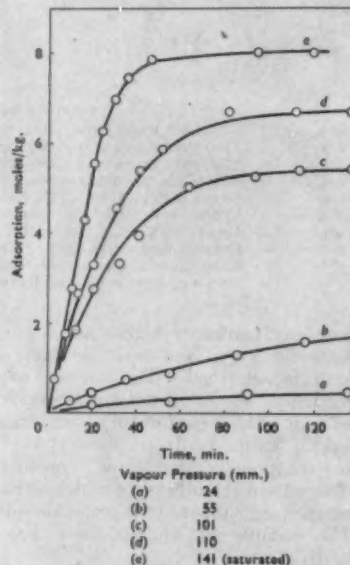


FIG. 5—Rates of Adsorption of Methanol Vapour on Wool at 27°C.

Fig. 5 shows some typical rate curves for methanol vapour on wool. At saturation vapour pressure, adsorption rises to 8 moles/kg.; this is equivalent to about 67% of the total amount of polar groups in wool, viz. peptide, hydroxyl, and amino and free acidic groups in the side-chains²⁶⁻²⁷. If the whole of this adsorbate is present as a monolayer in amorphous regions of the fibre, then about 23% of the fibre must be inaccessible

* There is evidence of 1:1-complex formation between an alcohol and an amide group in a dry solvent²¹.

to the methanol molecule. This figure compares favourably with values between 11% and 17% for the proportion of crystalline matter inaccessible to a smaller molecule, viz. deuterium oxide, in various types of wool fibre²⁸. On this result, therefore, it seems unnecessary to suppose that the adsorbate condenses into multilayers.

Plots of the first-order rate constants for methanol vapour adsorption on wool against vapour pressure (not shown) appeared to form two straight lines, intersecting at almost the same vapour pressure (95 mm.) at both 27° and 40°C., corresponding to adsorption values of 4.2 and 2.8 moles/kg. respectively; the significance of these values, if real, is not clear.

ADSORPTION OF INTENSIVELY DRIED ACETIC ACID

In order to determine whether traces of moisture cause significant changes in the adsorption of hydrophilic vapours on wool, the rate of adsorption

on wool of intensively dried glacial acetic acid was compared with that of normally dried acid. The former was found to have much the lower rate of adsorption (Fig. 6). Evidently the rate of diffusion of acetic acid in the fibre is lower than that of water, and its penetration is accelerated by the swelling effect produced by prior entry of traces of the more rapidly diffusing water vapour. The equilibrium adsorption of the normally dried acid vapour, like that of weak organic acids adsorbed from water²⁹, rises much higher than the value (ca. 0.85 mole/kg.) corresponding to the free amino groups, and no maximum adsorption was in fact observed in this test. Hydrogen bonding with the peptide groups and with some of the side-chain polar groups of the protein fibre must be the principal adsorptive force.

ADSORPTION OF DRY HYDROGEN CHLORIDE

Dry wool showed a barely measurable adsorption of dry hydrogen chloride gas (Table III, *l*), which

TABLE III
Adsorption Rate Data

| <i>t</i> | <i>C_F</i> | <i>t</i> | <i>C_F</i> | <i>t</i> | <i>C_F</i> | <i>t</i> | <i>C_F</i> | <i>t</i> | <i>C_F</i> | <i>t</i> | <i>C_F</i> |
|----------|----------------------|----------|----------------------|----------|----------------------|----------|----------------------|----------|----------------------|----------|----------------------|
| (a) | (b) | (c) | (d) | (e) | (f) | (g) | (h) | (i) | (j) | (k) | (l) |
| 2 | 373 | 9 | 1430 | 3 | 1231 | 1 | 6.8 | 2.5 | 267 | 1 | 518.2 |
| 7 | 398 | 16 | 1840 | 9 | 1991 | 2 | 54 | 4 | 356 | 2 | 883.1 |
| 11 | 633 | 20 | 2100 | 20 | 2646 | 5 | 122 | 8 | 445 | 4 | 1465 |
| 22 | 683 | 27 | 2310 | 50 | 3354 | 10 | 157 | 30 | 784 | 8 | 2274 |
| 32 | 795 | 30 | 2510 | 80 | 3981 | 30 | 206 | 60 | 1007 | 13 | 2698 |
| 52 | 907 | 50 | 3200 | 130 | 4540 | 65 | 327 | 157 | 1202 | 30 | 3570 |
| 80 | 1530 | 140 | 5830 | 210 | 5331 | 230 | 497 | 60 | 4220 | 80 | 4550 |
| 160 | 2030 | | | | | 260 | 875 | | | | |
| 215 | 2180 | | | | | | | | | | |
| 255 | 2830 | | | | | | | | | | |
| 1 | 716.0 | 1 | 978 | 6 | 65.8 | 1 | 96 | 1 | 221 | 3 | 0 |
| 2 | 1243 | 2 | 1443 | 11 | 97.3 | 5 | 128 | 3 | 547 | 6 | 15 |
| 4 | 1806 | 3 | 1809 | 20 | 230.3 | 22 | 179 | 7 | 918 | 10 | 30 |
| 8 | 2527 | 4 | 2079 | 30 | 342.2 | 32 | 288 | 10 | 1136 | 15 | 31 |
| 15 | 3123 | 6 | 2485 | 47 | 540.2 | 45 | 384 | 33 | 2077 | 30 | 15 |
| 30 | 3640 | 10 | 3081 | 60 | 704.1 | 75 | 512 | | | 270 | 30 |
| 60 | 4228 | | | | | 90 | 608 | | | | |
| 90 | 4438 | | | | | | | | | | |
| 1 | 68.6 | 1 | 185.7 | 1 | 955 | 1.5 | 1122 | 2 | 81 | 5 | 210 |
| 2 | 68.6 | 4 | 216.6 | 2 | 1179 | 2.5 | 1741 | 10 | 107 | 10 | 290 |
| 5 | 91.4 | 13 | 247.6 | 3 | 1321 | 4.5 | 2233 | 18 | 179 | 30 | 345 |
| 12 | 160 | 20 | 321.8 | 5 | 1565 | 9 | 3031 | 33 | 213 | 60 | 373 |
| 23 | 239 | 30 | 371.4 | 8 | 1749 | 10 | 3281 | 40 | 236 | 90 | 400 |
| 33 | 274 | 135 | 464.1 | 15 | 1992 | 12 | 3532 | 40 | 222 | 120 | 373 |
| | | | | 20 | 2093 | 15 | 3874 | 75 | 222 | | |
| | | | | 24 | 2113 | 20 | 4352 | 90 | 222 | | |
| | | | | | | 42 | 5607 | 162 | 264 | | |
| | | | | | | 75 | 5811 | | | | |

KEY

(Saturated vapour pressure in each case, except *l*)

- a, b, c* = Phenol, undrawn nylon, 60°, 70°, 80°C.
- d, e* = Acetic acid, undrawn, drawn nylon, 25°C.
- f, g, h* = Acetic acid, wool, 40-5°, 50-5°, 60-5°C.
- i, j* = Intensively dried acetic acid, wool, 35°, 45-5°C.
- k* = Intensively dried acetic acid, drawn nylon, 30°C.
- l* = Hydrogen chloride, wool, 25°C.; vapour pressure 78 mm. Hg. *t* = 10 hr. (see footnote* on p. 658)
- m* = β -Naphthol, undrawn nylon, 120°C.
- n* = β -Naphthol, wool, 130°C.
- o, p* = β -Naphthol, precipitated nylon, 133°, 140°C.
- q* = Aniline, drawn nylon, 70°C.
- r* = Ethylene glycol, drawn nylon, 100°C.

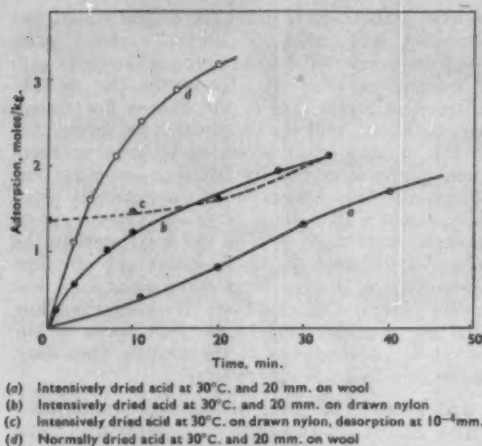


FIG. 6—Adsorption and Desorption Rates for Acetic Acid Vapour on Wool and Nylon

at normal temperatures should have very little van der Waals attraction for the fibre. Larose¹⁶ has reported very high values for the adsorption of carefully dried samples of this gas by wool (details of the drying and the degassing of the fibre are not given), and he attributes these to acid-base combination. Further investigation is obviously needed to correlate these apparently discordant series of results*.

ADSORPTION OF PHENOL VAPOUR

Rate curves were determined for the adsorption of phenol vapour (Fig. 7 and Table III, *a-c*). On nylon a slight discontinuity in the 25°C. rate curve is evident at 0.94 mole/kg. fibre; if two phenol molecules are attached to each amide group†, this value is equivalent to about 5.3% of the total number in the fibre. Thus the accessibility of amorphous regions is lower than for the smaller methanol molecule (see above), but the very high affinity of phenol ensures a subsequent rapid penetration of the crystalline regions, and the curve commences to rise again almost at once. At the higher temperatures the discontinuity is not discernible. The ultimate limit of adsorption on nylon would, of course, be indeterminate, since the fibre dissolves in phenol.

On wool the adsorption of phenol is slow, and the rate curves have plateaux, which increase in height and decrease in length with rise in temperature. Thus with rise in temperature the oriented regions of the fibre are becoming increasingly accessible. The steady state, during which little further adsorption appears to occur, must represent a slow coverage of the outer layers of the crystalline regions, leading to increase of the pressure gradient between outside and inside. The plateaux on the 25°C. and 60°C. curves are equivalent to an internal surface area of 3.5×10^5

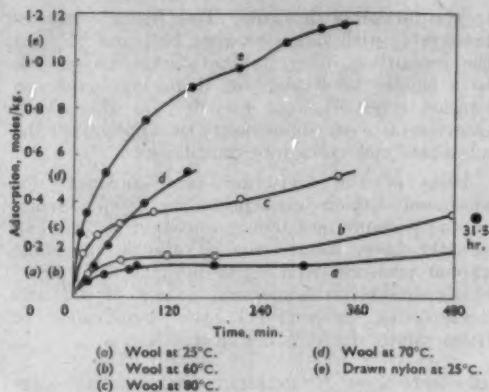


FIG. 7—Adsorption Rates of Saturated Phenol Vapour on Nylon and Wool

and 40×10^7 sq.cm. per gram of fibre respectively, assuming that two phenol molecules are attached to each peptide group.

HIGH-TEMPERATURE ADSORPTION TESTS

It was hoped that a greater variety of compounds might be tested as adsorbates if adsorption at high temperature were to prove practicable. Several compounds of high boiling point were in fact used successfully after it had been found that neither wool nor nylon appears to suffer damage when heated at temperatures even up to 150°C. for as long as 8 hr. at a pressure of 10^{-4} mm. of mercury. A loss in weight of about 12% and 4% respectively took place in the first hour of treatment, no doubt owing to degassing and removal of water, but no further change occurred. It was therefore concluded that these fibres are stable at this high temperature in complete absence of air and water‡.

Alizarin, benzoic acid, and β -naphthol were selected for trial in experiments at temperatures up to 140°C. Adsorption proceeded readily, and typical results are shown in Fig. 8 and 9 and Tables II (*a*)-(o) and III (*m*)-(p). The most striking effect revealed is the great and progressive increase in adsorption capacity of both nylon and wool in successive cycles of adsorption and desorption. This effect, so far as the authors are aware, has not previously been recorded, and must presumably be attributed to a progressive breakdown of the crystal structure of the fibres by disruption of internal hydrogen bonds and their replacement by new attachments to the bulky molecules of the adsorbate, or possibly to reduced chemical stability of the fibres in presence of these vapours. There is a maximum in the curve of the second adsorption cycle of benzoic acid on nylon, after which the amount adsorbed decreases with increase in pressure. This effect was found to be real and repeatable, but no explanation for it can be offered. It will be seen from Fig. 9 that the second, third, and fourth adsorption cycles on

* Note added in Proof—A recent repetition of the test here has given a much higher adsorption figure (ca. 2.2 moles/kg. at 22°C.), very much closer to Larose's.

† Gilles, Rose, and Vallance²⁰ detected a 2:1 complex between phenol and an amide group in dry benzene.

‡ Zahn²¹ states that dry wool is much more resistant to heat than wet wool, and it can safely be heated to 140–150°C. for a short period without damage, in absence of any substances which attack wool protein.

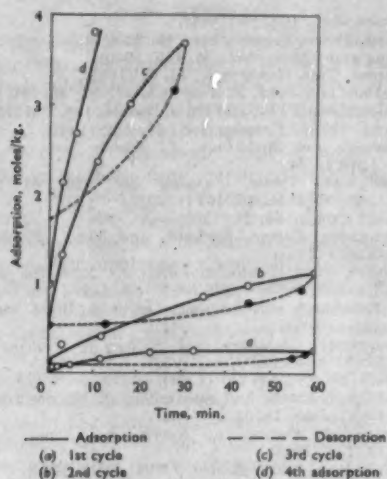


FIG. 8—Successive Adsorption-Desorption Cycles of β -Naphthol Vapour on Undrawn Nylon at 120°C.

wool more or less duplicate each other in rate of adsorption and in the additional amount of adsorbate taken up by the fibre in each cycle. In the case of undrawn nylon (Fig. 8), however, a great increase in both rate and maximum adsorbed is noticed with each successive cycle. This difference between the properties of the two substrates is no doubt due to the wool fibre being less readily swollen.

ENERGIES OF ACTIVATION

Table IV gives values of the energies of activation of a number of the vapours calculated from the initial slopes of the rate curves. These data are insufficient for any firm conclusions to be drawn, but it is clear that the values rise with increase in size of the hydrocarbon residue of the adsorbate, and that those for wool are somewhat higher than the corresponding ones for nylon, no doubt owing to the more firmly cross-linked structure of wool. The effect of the swelling action of traces of water in normally dried acetic acid is evident in its very low activation energy.

Conclusions

The main conclusions of this work are—

(a) Hydroxy compounds have much higher affinity for nylon and wool than have other polar organic compounds.

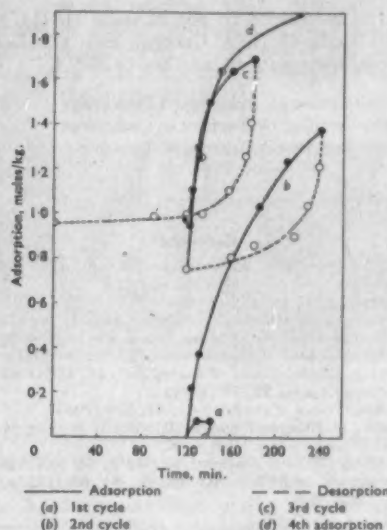


FIG. 9—Adsorption-Desorption Cycles of β -Naphthol Vapour on Wool at 120°C.

(b) The internal surface area available for monolayer formation in nylon is nearly the same for methanol and water, but much less for phenol, and the adsorption isotherms for methanol and water on nylon are identical, suggesting that the monolayer adsorption sites are also identical. They are probably the hydroxy groups of enolic amide or peptide groups¹.

(c) The crystalline structure of wool is increasingly broken down by phenols with rise in temperature (though not to the extent of complete dissolution).

* * *

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TABLE IV
Zero-point Energies of Activation

| Adsorbate | Fibre | $-\Delta E$ (approx.) (kcal./mole) |
|--------------------------------|----------------------|--|
| Acetic acid | Wool | (Table III; Fig. 6) <0.5 |
| Acetic acid, intensively dried | Wool | (Table III; Fig. 6) 4.7 |
| β -Naphthol | Nylon (precipitated) | (Table III) 5.3 |
| β -Naphthol | Wool | (Table III; Fig. 9) 7.4 |
| Phenol | Nylon (undrawn) | (Table III; Fig. 7) 5.0 |
| Phenol | Wool | (Fig. 7) 6.5 |

financial assistance to one of them (H.R.C.), and to the Delhi Cloth & General Mills Co. Ltd. for financial assistance to another (V.B.C.).

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The Rotproofing of Viscose Rayon

I—Treatment of Viscose Rayon with Formaldehyde and Formaldehyde-containing Resins

J. W. BELL, MARGARET M. RAMSEY, and C. S. WHEWELL

Viscose rayon yarn is treated with formaldehyde and a number of formaldehyde-containing resins in an attempt to obtain a rotproof finish. The most outstanding result which emerges from the experiments is that halogenated phenoplasts are more efficient than either urea-formaldehyde or phenol-formaldehyde, and that a resin prepared from bromophenol and formaldehyde is exceedingly effective, a deposit of only 5% on the yarn being resistant to microorganisms under the most severe and prolonged test conditions. It also appears possible to improve the efficiency of urea-formaldehyde resin by incorporating a small proportion of halogenated resin.

Urea-formaldehyde resin by itself is effective only if present in prohibitively high concentrations; below 15–20% the proof breaks down on prolonged testing. Formaldehyde alone is moderately effective, and appears to have little effect on the wet tensile strength of the viscose rayon; it may be of value where conditions highly favourable to the growth of microorganisms are not likely to be encountered.

The experiments have shown that laboratory assessment of rotproofing should be carried out by more than one method; a treatment which confers a measure of resistance to microorganisms under one set of test conditions may be much less effective when tested by a different procedure. This confirms the findings of many other workers.

Cellulosic fibres are widely used in the manufacture of fabrics which are destined to be employed under conditions entailing exposure to attack by microorganisms. Wastage due to rotting reaches particularly serious proportions in tropical climates, and materials such as tentage, cotton uniforms, and camouflage netting have in recent years been extensively employed under the most adverse conditions possible. In consequence a great deal of research has been directed to discovering methods of proofing cellulosic textiles against fungi and bacteria. Most of this work has been concerned with cotton, and a very thorough survey of the progress made has been given by Siu¹. The application of rotproofing treatments to regenerated cellulose rayons does not, however, appear to have received much attention. It is clearly desirable that any process should be easily accommodated within the existing manufacturing

routines, and one which comes readily to mind is that of resin treatment. Crease-resist processes are well known and, as it has been claimed^{2–4} that the presence of aminoplasts on cotton cellulose inhibits the growth of microorganisms, it was decided to study the effect of these compounds on the resistance of viscose rayon to microorganisms.

PREPARATION OF MATERIALS

Continuous-filament viscose rayon yarn (standard S twist, 500 denier, 50 filaments, supplied by Messrs. Courtaulds Ltd.) was used throughout. Hanks weighing approx. 2 g. (air-dry) were prepared, and purified in a Soxhlet extractor for 8 hr. with ether and 8 hr. with absolute alcohol. They were then washed in running tap-water (24 hr.) and in three changes of distilled water (3 × 8 hr.), dried in air, and conditioned at 22–2°C. and 65% R.H.

TEST METHODS

Rotproofed textiles are usually tested for resistance to microorganisms by exposing a sample to suitably destructive conditions and subsequently assessing the strength or serviceability of the material. Owing to the impracticability of field trials, e.g. soil burial or outdoor weathering, laboratory methods were employed in this investigation. Three types of test procedure were selected. In the first, a pure culture of one organism was used in a medium which provided all the necessary nutrients except carbon, this being present in the viscose rayon under test. In the second, the material was placed in contact with horse-dung, which itself contained all the nutrients necessary to maintain a flourishing growth of many types of organism⁵. This is a particularly severe test, as there is always the possibility that material in the dung preparation may react with the proofing agent on the yarn, thus opening the way to attack by the microorganisms. The third procedure was a combination of the other two, the yarn being moistened with a liquid horse-dung extract⁶ under conditions permitting free access of air. All the tests were carried out in an incubator at $30 \pm 1^\circ\text{C}$. and in an atmosphere saturated with water vapour. For the sake of brevity, this procedure is referred to as *incubation*.

Incubation in a Pure Culture

For this work the organism *Chaetomium globosum* was selected, being preferred to more vigorous organisms such as *Myrothecium verrucaria* because it is widespread in Nature, easy to manipulate in the laboratory, and less liable to develop variants⁵.

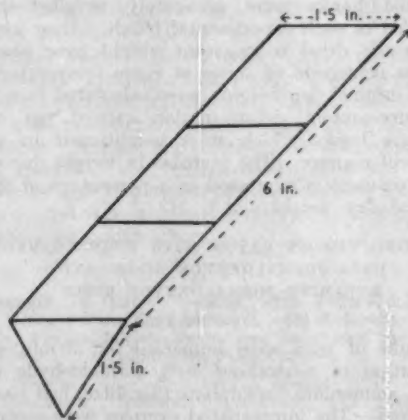


FIG. 1

The complete procedure was as follows—

Circles approx. 4 cm. in diameter were cut from the centre of the metal cap in each of a number of 20-oz. glass jars, and replaced by circles of Tyglas* fabric. Glass frames (Fig. 1 and 2) were constructed from $\frac{1}{8}$ in. diameter glass rod in such a fashion that they would lie well above the surface

of the nutrient medium which the jar, lying horizontally, would subsequently contain: one end of the frame rested on the neck of the jar, and the end with the triangular projection was supported on the inside of the jar.

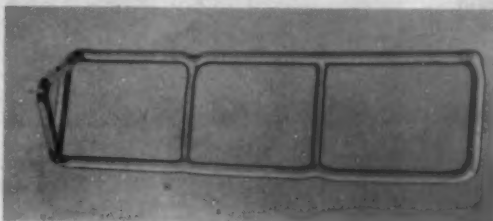


FIG. 2

Spore suspensions of *C. globosum* were prepared by shaking an actively sporing culture, grown on dilute potato dextrose agar, with sterile glass beads until examination under the microscope indicated a uniform suspension. A nutrient medium was prepared containing—

| | g./litre |
|---|----------|
| Dipotassium hydrogen phosphate, K_2HPO_4 | 1.394 |
| Magnesium sulphate, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ | 0.74 |
| Ammonium nitrate, NH_4NO_3 | 1.00 |
| Sodium chloride, NaCl | 0.005 |

the initial pH value being 6.8. To each hank of viscose rayon yarn were sewn two strips (7 in. \times 0.5 in.) of Tyglas fabric, one at each end (Fig. 3); 50 ml. of nutrient medium was placed in each 20-oz. glass jar, and the hanks, jars, and glass frames were sterilised separately at 15 lb./sq.in. pressure for 20 min. Each hank was then inoculated by spraying it uniformly with 5 ml. of the spore suspension, and two hanks were laid lengthwise on each frame. The frames were inserted in the jars so that the glass strips dipped into the nutrient medium; photographs of the assembly are shown in Fig. 4 and 5. The jars were placed on their sides in an incubator at $30 \pm 1^\circ\text{C}$., the atmosphere being maintained saturated with

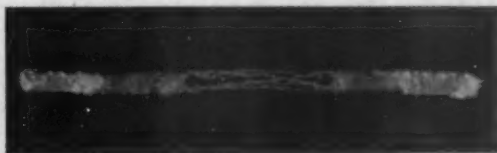


FIG. 3

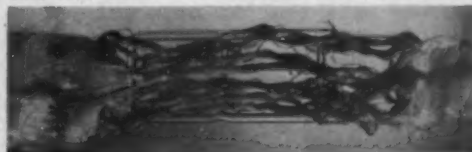


FIG. 4

* Fothergill & Harvey Ltd.

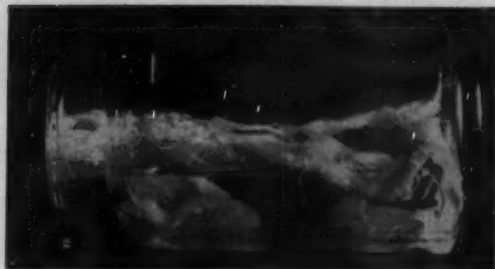


FIG. 5

water vapour. Batches of four hanks were removed at selected intervals of time for testing, and if after 28 days there appeared to be no appreciable loss in yarn strength or no visible fungoid growth on the hanks, they were re-inoculated with 5 ml. of spore suspension, and the nutrient medium was renewed.

Incubation in Contact with Horse-dung Preparation

About 200 g. of horse-dung (freshly gathered from a country field) was mixed to a sludge with 1250 ml. of tap-water, and filtered through surgical gauze. The residue was allowed to drain (without suction) for 10 min., after which it contained about 30% more moisture than that originally present. This quantity was sufficient to fill 14 Petri dishes (3½ in. in diameter) to a depth of 0.25–0.5 in. One hank of yarn was laid spirally on the surface of the dung in each dish, and pressed firmly on. The lids were placed in position, and the dishes incubated at $30 \pm 1^\circ\text{C}$. in an atmosphere saturated with water vapour. Once a week the dung was moistened with 5 ml. of water, care being taken not to run the water directly on the yarn. Batches of four hanks were removed at selected intervals of time for testing.

Incubation with Horse-dung Extract

Incubation in contact with solid horse-dung suffers from a marked defect in that air is to a large extent excluded from the under-surface of the hanks. Since, under test conditions, microbial attack is greatest when there is free access of air, this defect results in uneven degradation of the cellulose. A procedure was therefore adopted similar to that used in the pure-culture experiments. Two-gram hanks of rayon, folded to a length of 4 in., and with a glass wick (7 in. \times 0.5 in.) sewn to either end, were inoculated with 5 ml. of the liquid extract obtained in preparing the sludge for the contact tests. Two hanks were laid lengthwise on a glass frame, in the manner described above, and the frame was placed in a horizontal 20-oz. glass jar containing 50 ml. of extract, into which the glass strips dipped. The jar caps, with air-permeable lids, were screwed on, and the jars incubated at $30 \pm 1^\circ\text{C}$. in an atmosphere saturated with water vapour. At selected intervals of time batches of four hanks were removed for testing. If no appreciable loss in yarn breaking

strength had occurred after 28, 65, and 100 days, the hanks were re-inoculated and the extract was renewed.

To check the viability of the organisms in the medium (all three methods) in instances where, after prolonged incubation, no loss in tensile strength was found, cultures were taken at random from the suspension and/or test samples, plated on to a malt agar (malt extract 15 g., agar 25 g., water 1 litre) and nutrient agar (Lab-lemco (Oxo) 10 g., peptone 10 g., sodium chloride 5 g., water 1 litre; pH value 7.0), incubated at $30 \pm 1^\circ\text{C}$., and examined for growth at frequent intervals. No attempt was made to identify the organisms or to assess the amount of growth.

METHOD OF ASSESSING DAMAGE

Damage caused by the treatments applied was assessed by measuring the tensile strength of the wet yarn. Determinations were carried out on a Baer single-thread tester, using a short test length; unless otherwise stated, values recorded are each the mean of 125 breakages. Results are expressed as the percentage residual strength; if a proofing treatment was applied the 100% value was that of the proofed yarn. Where necessary, a separate account is presented of the change in strength caused by the treatment.

Each proofing treatment was tested by all three methods, and in all instances unproofed hanks were incubated at the same time.

MEASUREMENT OF THE AMOUNT OF RESIN DEPOSITED ON THE FIBRE

To ascertain the amount of resin deposited on the yarn in any individual treatment, three conditioned hanks were accurately weighed and included in each experimental batch. They were afterwards dried to constant weight over phosphorus pentoxide *in vacuo* at room temperature. Their original dry weights were calculated from a moisture-content determination carried out on separate hanks which were conditioned in an identical manner. The increase in weight due to the treatment is expressed as a percentage of the original dry weight.

TREATING VISCOSE RAYON WITH FORMALDEHYDE, UREA-FORMALDEHYDE RESIN, AND MELAMINE-FORMALDEHYDE RESIN

(1) Formaldehyde

Hanks of yarn were immersed for 30 min. in a solution of neutralised 20% formaldehyde in which ammonium persulphate (3 g./litre) had been dissolved. The impregnated samples were centrifuged for 5 min., dried in air, and heated at 120°C . for 5 min. They were then washed in water containing 0.5% of Teepol (Shell) and 0.25% of ammonia at 80°C . for 5 min., rinsed well in water, and dried. An increase in weight of 1.6% was obtained.

(2) Melamine-Formaldehyde Resin

A modified melamine-formaldehyde resin (Beetle Textile Resin BT 309) was used. A 10% solution (wt./vol.) of resin dope, containing 10% of

Accelerator AC 15 (a 20% solution of ammonium thiocyanate) and 10% of wetting agent (Teepol) on the weight of resin, was prepared. Samples of yarn were immersed in this dope for 15 min., centrifuged for 5 min., dried in air, and cured at 140°C. for 3 min. They were then scoured at 50°C. in water containing 0.5% of Teepol and 0.25% of ammonia, rinsed well, and dried. An increase in weight of 6.0% was obtained.

(3) Urea-Formaldehyde Resin

Urea-formaldehyde monomer was prepared by dissolving 100 g. of urea in 200 ml. of 40% formaldehyde which had been brought to pH 7.0 ± 0.2 by adding dilute sodium hydroxide solution. To the solution was then added 9 ml. of ammonia (sp.gr. 0.880), thus bringing the pH value to about 9. The solution, which had a density of 28–31°Tw., was allowed to stand at room temperature for about 12 hr., at the end of which time the density had increased to about 35°Tw. By diluting with tap-water a dope of density 7°Tw. was prepared. Before use, 10 ml. of 10% ammonium dihydrogen phosphate per 100 ml. of dope was added as a catalyst. The final pH value was approx. 4. Hanks of viscose rayon were wetted out for 15 min. in distilled water, centrifuged for 5 min., impregnated in the urea-formaldehyde dope for 8 min., centrifuged for 5 min., dried in air, and cured for 5 min. at 137°C. They were then scoured in water containing 0.5% of Teepol and 0.25% of ammonia (sp.gr. 0.880) at 80°C. for 5 min., after which they were rinsed well and dried in air. An increase in weight of 5% was obtained.

Results of strength tests carried out on the yarns after incubation by the three methods are given in Tables I–III.

These results show clearly that the three incubation methods differ widely in severity, and also that a proof which stands up reasonably well to one procedure is virtually useless against another. Nor are the relative severities constant; for example, the resin-treated yarns are least severely degraded by the pure-culture test, whereas the control yarns are least severely degraded by the horse-dung contact test.

TABLE I

Strength of Yarns treated with Formaldehyde, Urea-Formaldehyde Resin, and Melamine-Formaldehyde Resin, and incubated in *C. globosum* Pure Culture

| Time of Incubation (days) | Residual Strength (%) ^a | | | |
|---------------------------|------------------------------------|--------|--------|------------|
| | No Proof | 6% m-f | 5% u-f | 1.6% H-CHO |
| 0 | 100 | 100* | 100* | 100* |
| 4 | 59.0 | 109.4 | 109.0 | 111.0 |
| 7 | 30.0 | 99.4 | 113.0 | 95.4 |
| 14 | 15.8 | 113.0 | 74.4 | 89.0 |
| 21 | 14.7 | 110.4 | 41.4 | 77.0 |
| 28 | 11.4 | 104.0 | 26.7 | 75.2 |
| 42 | 0.0 | 79.6 | 19.3 | 77.0 |
| 63 | — | 54.4 | 4.5 | 52.3 |

^a All strengths are calculated as a percentage of the proofed yarn before incubation.

m-f = Melamine-formaldehyde
u-f = Urea-formaldehyde

TABLE II

Strength of Yarns treated with Formaldehyde, Urea-Formaldehyde Resin, and Melamine-Formaldehyde Resin, and incubated in Contact with Horse-dung

| Time of Incubation (days) | Residual Strength (%) | | |
|---------------------------|-----------------------|--------|--------|
| | No Proof | 6% m-f | 5% u-f |
| 0 | 100 | 100 | 100 |
| 4 | 93.8 | 86.6 | 92.5 |
| 7 | 72.3 | 81.3 | 74.0 |
| 14 | 35.3 | 45.9 | 63.0 |
| 21 | 27.1 | 25.2 | 48.8 |
| 28 | 20.0 | 11.9 | 34.4 |
| 42 | 16.4 | 0.0 | 26.2 |
| 63 | 0.0 | — | 15.1 |

TABLE III

Strength of Yarns treated with Formaldehyde, Urea-Formaldehyde Resin, and Melamine-Formaldehyde Resin, and incubated in Horse-dung Liquid Extract

| Time of Incubation (days) | Residual Strength (%) | | |
|---------------------------|-----------------------|--------|--------|
| | No Proof | 6% m-f | 5% u-f |
| 0 | 100 | 100 | 100 |
| 4 | 69.3 | 84.8 | 98.8 |
| 7 | 53.8 | 55.6 | 83.7 |
| 14 | 35.4 | 28.7 | 67.7 |
| 21 | 17.0 | 19.8 | 41.1 |
| 28 | 11.3 | 11.6 | 35.1 |
| 45 | 0.0 | 6.6 | 18.5 |
| 65 | — | 0.0 | 13.7 |
| 100 | — | — | 2.2 |
| 140 | — | — | 0.0 |

Similar variations are apparent when the proofs are compared. Melamine-formaldehyde is distinctly better than urea-formaldehyde when tested with a pure culture, but is slightly inferior when tested by the other two methods. The results obtained with formaldehyde-treated yarn are particularly interesting, being very much better than urea-formaldehyde resin when tested by all three methods and than melamine-formaldehyde when tested by the horse-dung contact and liquid extract methods.

A weight increase of 6.0% of melamine-formaldehyde resin corresponds approximately to 1 molecule of monomer per 22 anhydroglucose units. If, as postulated by Cooke³, the monomer reacts with three adjacent cellulose chains to form a triether, the ratio of monomer molecules to etherified anhydroglucose units is about 1:7. A ratio of 1:7 is obtained also with a deposit of 5% of urea-formaldehyde. An increase in weight of 1.6% due to formaldehyde alone, however, corresponds to a ratio of 1 methylene group per 2.7 anhydroglucose units, i.e. a reacted ratio of roughly 1:1.5. This much higher proportion of reacted anhydroglucose units may be the reason for the superiority of the formaldehyde treatment. On the other hand, the two resins differ considerably in their proofing effect, despite their reacting in similar proportions according to the above argument.

In short, the main conclusion is that deposits of 6% of melamine-formaldehyde and 5% of urea-formaldehyde resins are ineffective against prolonged incubation with microorganisms.

It is of interest to record the changes in yarn strength which occur as a result of the proofing treatment. Data are given in Table IV.

| Proof | Strength (% of original) |
|-------------------------------------|-----------------------------|
| Formaldehyde (1.6%) | 96.4 |
| Urea-formaldehyde (5.0%) | 138.4 |
| Melamine-formaldehyde (6.0%) | 131.8 |

The small loss in strength of formaldehyde-treated viscose rayon is of particular interest, as similar treatment of cotton may result in a considerable loss in strength, the amount depending on the reaction conditions⁶. Goldthwait⁷ cites a treatment in which cotton lost approximately half of its breaking strength by the time a weight increase corresponding to 2.64% of combined formaldehyde was attained. The treated material, however, lost only 20% of its strength after 11 weeks' soil burial. Marsh⁸ has stated that it does not appear to be possible to obtain crease-resisting effects by formation of methylenecellulose without substantial degradation, the damage to native cellulose being greater than that to regenerated cellulose.

As such a loss in strength is brought about by the treatment with formaldehyde, it is possible that practical use could be made of this method in instances where the subsequent service conditions are not very severe.

Small concentrations of resin having been comparatively ineffective, it was decided to apply different amounts of one of them, urea-formaldehyde being selected.

EFFECT OF DIFFERENT AMOUNTS OF UREA-FORMALDEHYDE RESIN

A solution of urea-formaldehyde precondensate was prepared as described above, and diluted with tap-water to provide a series of solutions with densities of 7, 10, 16, 20, and 24°Tw. After addition of catalyst solution, batches of hanks were impregnated, cured, and scoured as previously described. Increases in weight of 5, 8, 12, 18, and 22% were obtained. Samples were incubated by all three methods, and the results of subsequent strength tests are given in Table V. For the sake of brevity, only the figures for horse-dung contact tests are quoted.

It is clear that, although urea-formaldehyde resin affords some protection at as low a concentration as 5%, complete protection is not achieved even when 22% of resin is present in the fibre, though there is a steady increase in resistance as the weight of resin increases. In all the tests the yarn treated with 22% of resin substantially retains its strength up to about 45 days, but, as seen from Table V, there is a loss in strength of 80% after 140 days in horse-dung extract, a result

TABLE V
Strength of Yarns proofed with Different Amounts of Urea-Formaldehyde Resin and tested by the Horse-dung Extract Method

| Resin deposited, %— | 0 | 5 | 8 | 12 | 18 | 22 |
|---------------------------|-----------------------|------|-------|-------|------|-------|
| Time of Incubation (days) | Residual Strength (%) | | | | | |
| 0 | 100 | 100 | 100 | 100 | 100 | 100 |
| 4 | 69.3 | 98.8 | 100.8 | 100.7 | 97.5 | 103.1 |
| 7 | 53.8 | 83.7 | 99.2 | 102.7 | 90.8 | 109.4 |
| 14 | 35.4 | 67.7 | 72.8 | 109.7 | 81.1 | 97.9 |
| 21 | 17.0 | 41.1 | 70.5 | 98.1 | 86.8 | 108.4 |
| 28 | 11.3 | 35.1 | 42.2 | 96.2 | 62.8 | 104.7 |
| 35 | — | — | — | — | 53.0 | — |
| 42 | — | — | — | — | 58.5 | — |
| 45 | 0.0 | 18.5 | 27.1 | 79.3 | — | 102.1 |
| 65 | — | 13.7 | 14.7 | 74.4 | — | 78.0 |
| 100 | — | 2.2 | 4.5 | 35.7 | — | 89.0 |
| 140 | — | 0.0 | 1.5 | 19.5 | — | 20.9 |

no better than when 12% of resin is present. It is possible that slowly growing organisms, against which urea-formaldehyde is ineffective, have had time to develop; alternatively, the treatment may not be uniform. The pure culture and horse-dung contact tests were carried on for up to 63 days only, at the end of which time the samples treated with only 12% of resin or less had lost 50% or more of their strength, compared with only 10% loss for the samples containing 22% of resin.

On the assumption that bishydroxymethylurea forms a diether with two adjacent cellulose chains, the ratio of resin molecules to molecules of reacted anhydroglucose units is approx. 1:1 when 22% of resin has reacted with the cellulose.

Changes in strength of viscose rayon yarns treated with different weights of urea-formaldehyde resin are recorded in Table VI.

TABLE VI
Changes in Strength of Viscose Rayon Yarns treated with Different Concentrations of Urea-Formaldehyde

| Resin (% by wt.) | Strength of Yarn (% of original) |
|---------------------|-------------------------------------|
| 5 | 138.4 |
| 8 | 135.9 |
| 12 | 136.3 |
| 18 | 84.1 |
| 22 | 98.0 |

It is clear that the amount of urea-formaldehyde resin necessary to confer on viscose rayon a satisfactory resistance to microorganisms is too great to be of practical value. A further series of experiments was therefore carried out in which much smaller quantities of resin were applied together with copper sulphate, copper naphthenate, and copper tartrate.

TREATMENT WITH COPPER SULPHATE AND UREA-FORMALDEHYDE

As a fairly efficient proof was obtained by Furry *et al.*⁹ when using a copper sulphate and wax-aluminium acetate emulsion, it was decided to attempt a similar copper sulphate treatment in conjunction with an 8% deposit of urea-formaldehyde resin.

sodium hydroxide solution. On rapid cooling the solution separated into two layers, the lower one being run off and diluted with an equal volume of 40% formaldehyde. Hanks of yarn were impregnated in this solution for 30 min., centrifuged for 5 min., dried, cured at 180°C. for 5 min., scoured, rinsed, and dried. A weight increase of 4.9% was obtained.

The above treatments resulted in deposits of resin which, by virtue of their different weights, are not strictly comparable. By adjusting the concentrations of the resin preparations a series of comparable treatments were carried out, and samples of yarn treated by all methods were tested by the three incubation procedures. Results from the different tests were very similar, those for the horse-dung liquid extract method being given in Table VII.

It is clear that the presence of a halogenated resin leads to a remarkable improvement in resistance to microorganisms, a deposit of only 5% of bromophenol-formaldehyde being 100% effective even after 140 days' incubation. Chlorophenol-formaldehyde, though very much better than phenol-formaldehyde, is not so effective as bromophenol-formaldehyde; the 18% deposit which appears necessary for 100% protection is too great to be of practical value.

All the phenolic resins were found to colour the yarn a light yellowish brown, but they had no adverse effect on the yarn strength, which in all instances was slightly increased.

THE EFFECT OF MIXING UREA-FORMALDEHYDE AND HALOGENATED PHENOL-FORMALDEHYDE RESINS

Parallel with the experiments described above, a series of trials was made using a urea-formaldehyde resin preparation in which was incorporated a small proportion of chlorophenol-formaldehyde. This, rather than the more effective bromophenol-formaldehyde, was selected because the results described above were not available when the experiment was started.

The impregnating solution was prepared as follows—

320 ml. of 40% formaldehyde solution, 128 g. of *o*-chlorophenol, and 12.8 ml. of sodium hydroxide solution (63°Tw.) were mixed and boiled under a reflux condenser for 15 min. The solution was cooled rapidly and diluted with an equal volume of 40% formaldehyde. Urea-formaldehyde monomer was prepared as previously described, and to it was added a volume of 40% formaldehyde equal to the volume of water required to bring the density to 10°Tw. The two monomer solutions were then mixed in the ratio of 1 part by volume of chlorophenol-formaldehyde to 4 parts by volume of urea-formaldehyde. A solution of catalyst was prepared by dissolving 3.71 g. of boric acid and 14.84 g. of tartaric acid in 100 ml. of distilled water, and was added to the mixed monomers to give a 1.6% (by vol.) solution, the final pH value being approx. 4.

Hanks of viscose rayon yarn were impregnated with the resin solution for 30 min., centrifuged for

5 min., dried, cured at 175°C. for 5 min., scoured, rinsed, and dried. An increase in weight of 15% was recorded. Samples were incubated by the pure culture and liquid horse-dung extract methods up to a maximum time of 42 days. Results of the horse-dung extract test are shown in Table VIII, in which values obtained on yarn treated with 12% urea-formaldehyde alone (with a different catalyst) are also included.

TABLE VIII

Residual Strength (%) of Yarns treated with Mixed Resins and tested by the Horse-dung Extract Method

| Time of Incubation (days) | Control (no resin) | 12% Urea-Formaldehyde | 15% Mixed Resins |
|---------------------------|--------------------|-----------------------|------------------|
| 0 | 100 | 100 | 100 |
| 4 | 69.3 | 100.7 | 99.9 |
| 7 | 53.8 | 102.7 | 99.5 |
| 14 | 35.4 | 109.7 | 87.7 |
| 21 | 17.0 | 98.1 | 83.6 |
| 28 | 11.3 | 96.2 | 89.0 |
| 35 | — | — | 97.0 |
| 42 | 0.0 | 79.3* | 114.4 |

* 45 days

Exact comparison is not possible between results obtained with 15% of mixed resins and 12% urea-formaldehyde alone, as the data given in Table V have shown that there is a considerable increase in proofing efficiency as the weight of resin increases from 12% to 18%. The most interesting aspect of the results is the great improvement in long-term resistance; in both methods of test there is a substantial increase in strength after 21 days' incubation. If this is a permanent effect, the incorporation of halogenated phenoplasts into urea-formaldehyde resin may be of considerable value.

Yarn treated with the mixed resins had a yellow colour and, in marked contrast to the previous results, lost about 20% of its strength as a result of the treatment.

PREPARATION OF RESINS FROM HIGHLY SUBSTITUTED CHLOROPHENOLS

As it has been claimed¹⁵ that increasing the halogen substitution of phenolic compounds leads to an increase in their antibacterial properties, attempts were made to prepare resins from formaldehyde and chlorophenols having a degree of substitution greater than unity. These experiments, using 2,4-dichlorophenol, 2,4,6-trichlorophenol, and pentachlorophenol, were unsuccessful, no polymer being formed; this was not unexpected, because the formation of phenoplasts depends on certain positions in the benzene ring being free for substitution reactions.

One of us (M.M.R.) wishes to acknowledge the grant of a scholarship from the British Rayon Research Association.

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(Received 13th June 1955)

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CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Tests for Fastness to Washing

In this year's examination for the A.S.D.C. one question (Paper E (i) 8) asked the candidate to describe the Society's methods for testing the fastness to washing of coloured materials¹.

In the report of the Diplomas Executive Subcommittee² the comment is made that: "One paper described the Society's methods using the Marney machine instead of the Wash Wheel", the implication being, of course, that the candidate was in error in so doing.

Although at the time of the examination the Society's methods were officially restricted to the Wash Wheel, two weeks later the position was completely changed with the publication of the *Third Report of the Fastness Tests Co-ordinating Committee*³, which permits the use of suitable mechanical washing devices such as the Marney machine for many of the present S.D.C. washing tests.

The purpose of this letter is not, however, to criticise the examiners' published comments (which,

although perhaps unnecessary, were just) nor to compliment the candidate on his intelligent anticipation of the Society's decision (which was probably unintentional), but to point out that the use of mechanical devices other than the Wash Wheel is now generally permitted. Marney machines have been used continuously for washing tests in this laboratory for the past seven years, and attachments for this purpose have been described⁴.

K. McLAREN

DYEHOUSE DEPARTMENT
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23rd September 1955

¹ *J.S.D.C.*, **71**, 492 (Sept. 1955).

² *J.S.D.C.*, **71**, 495 (Sept. 1955).

³ *J.S.D.C.*, **71**, 283 (June 1955).

⁴ *Hexagon Digest*, (14), 26 (1953).

Notes

Meetings of Council and Committees
October

Council—19th
Publications—18th
Colour Index Editorial Panel—5th
Fastness Tests Co-ordinating—14th
Perkin Centenary—3rd
Perkin Executive—28th
Perkin Publicity—21st
Review of Textile Progress—24th

Deaths

Mrs. Helena Smith, wife of Mr. Fred Smith, died on October 26th. The President will know that he has the deep sympathy of all members of the Society in his sad bereavement.

We regret to report the loss by death of Mr. C. L. Littlefair.

Perkin Centenary Celebration 1956

The names of those chiefly concerned in organising the Celebration are as follows—

Patron

H.R.H. The Duke of Edinburgh, K.G., K.T.,
F.R.S.

President

The President of the Royal Society

Vice-presidents

The President of The Society of Dyers and
Colourists

The President of The Chemical Society

The President of The Society of Chemical
Industry

The President of The Royal Institute of Chemistry
 The Chairman of the Association of British Chemical Manufacturers
Chairman of The Perkin Centenary Celebration Committee
 Sir Robert Robinson, O.M., F.R.S.
Chairman of The Executive Committee
 C. J. T. Cronshaw, Esq., D.Sc.
 (*Deputy Chairman*—
 C. M. Whittaker, Esq., D.Sc.)
Chairman, Finance Committee
 H. Jackson, Esq.
Chairman, Technical and Scientific Committee
 Professor W. Bradley, Ph.D., D.Sc.
Chairman, Social Committee
 Sir Ernest Goodale, C.B.E., M.C.
Chairman, Publicity Committee
 Richard J. Smith, Esq.
Bankers
 Martins Bank Ltd., Queens Road Branch, Manchester
General Secretary
 John W. Nicholls, Esq., F.C.I.S.

Bradford Junior Branch

The address of the Honorary Secretary, Mr. N. G. Morton, is now 22 Ashbourne Grove, Halifax.

The Worshipful Company of Dyers

Mr. Arthur H. Brewin has been elected Prime Warden. The new Renter Warden is Mr. H. B. Sissmore.

The Worshipful Company of Feltmakers

The following officers of the Worshipful Company of the Art or Mystery of Feltmakers of London have been elected for the ensuing year—

Master

Mr. Ernest Bright Laycock, O.B.E.

Upper Warden

Lt.-Col. John A. Christie-Miller, O.B.E.

Renter Warden

Dr. T. Collyer Summers, M.B., B.S., F.R.C.S.

Third Warden

Sir Heneage Ogilvie, K.B.E., M.C., F.R.C.S.

Fourth Warden

Mr. Philip Keens, F.C.A.

Clerk

Mr. Sidney Alfred Williamson

Deputy Clerk

Mr. Ian David Wilson.

B.S.I. Sales Office in Birmingham

Through the co-operation of the Birmingham Chamber of Commerce, the British Standards Institution has now opened a Sales Office in the headquarters of the Chamber at 95 New Street, Birmingham 2. The new office in Birmingham will maintain a full and up-to-date stock of all British Standards and associated publications.

Effect of Colour on Apparent Size

Work carried out in co-operation between the Pittsburgh Plate Glass Co. and the psychological laboratory of Johns Hopkins University shows that change in hue alone of an object can effect at least a 9.5% change in apparent size, change in brightness an 8.5% change, and change in chroma or saturation a 6% change. Combined in the same direction these three factors can cause an apparent change of 13.5%. It is not possible to say that e.g. red makes things appear larger whereas if they are blue they appear smaller, but specific reds and blues must be referred to. On the whole, the longer the wavelength or the greater the chroma the larger the apparent size of the object. C.O.C.

New Books and Publications

Case Study Data on Productivity and Factory Performance

Cotton Textile Dyeing and Finishing

Bureau of Labor Statistics Report No. 66

Prepared for the Foreign Operations Administration by the U.S. Department of Labor. Pp. vii + 151. (London: British Institute of Management.) 1954. Price, 7s. 6d.

This book furnishes in great detail the results of an enquiry into the production of seven cotton dyeing and finishing plants in the U.S.A., undertaken by the U.S. Department of Labor for the Industrial and Technical Assistance Division of the Foreign Operations Administration. It is intended to provide material for comparison by European production managers and methods engineers

between their own works and the same operations or processes in one or more of the plants surveyed. There is a clear and concise examination of processes and a wealth of statistical information, particularly on man-hours expended per unit of fabric production.

Specialisation in types of fabrics and processes applied and the use of high-speed continuous machinery are claimed to account largely for the level of production in the U.S. dyeing and finishing industry. Many plants specialise in bleached and mercerised white goods. Others concentrate on much more complex production.

The seven plants providing the case studies vary considerably, and none is claimed as typical of the U.S. industry. Only three were designed



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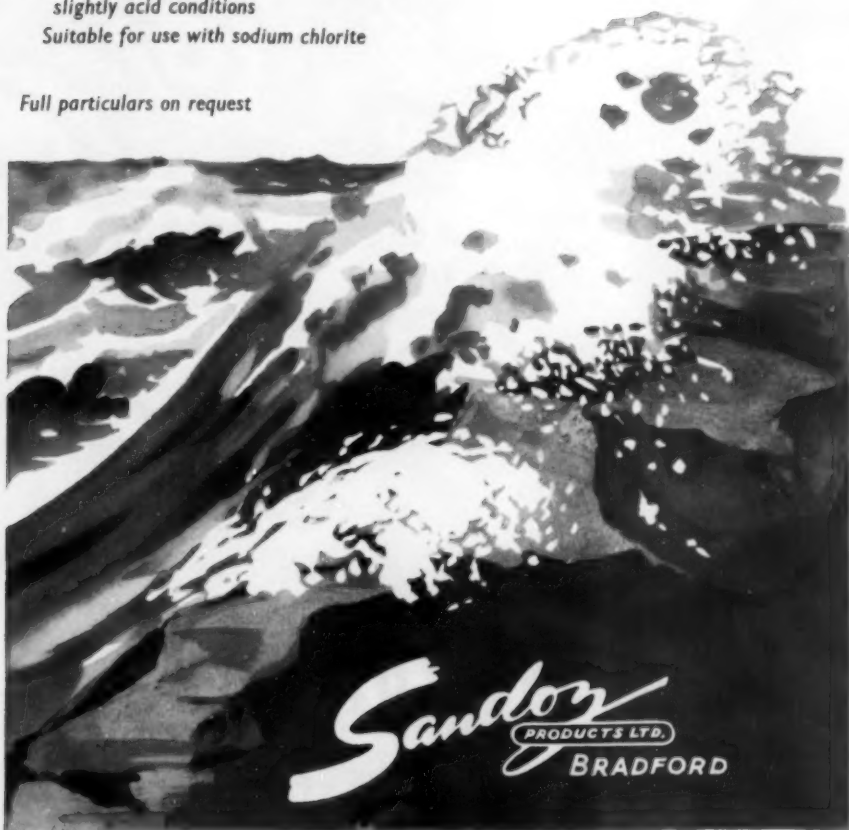
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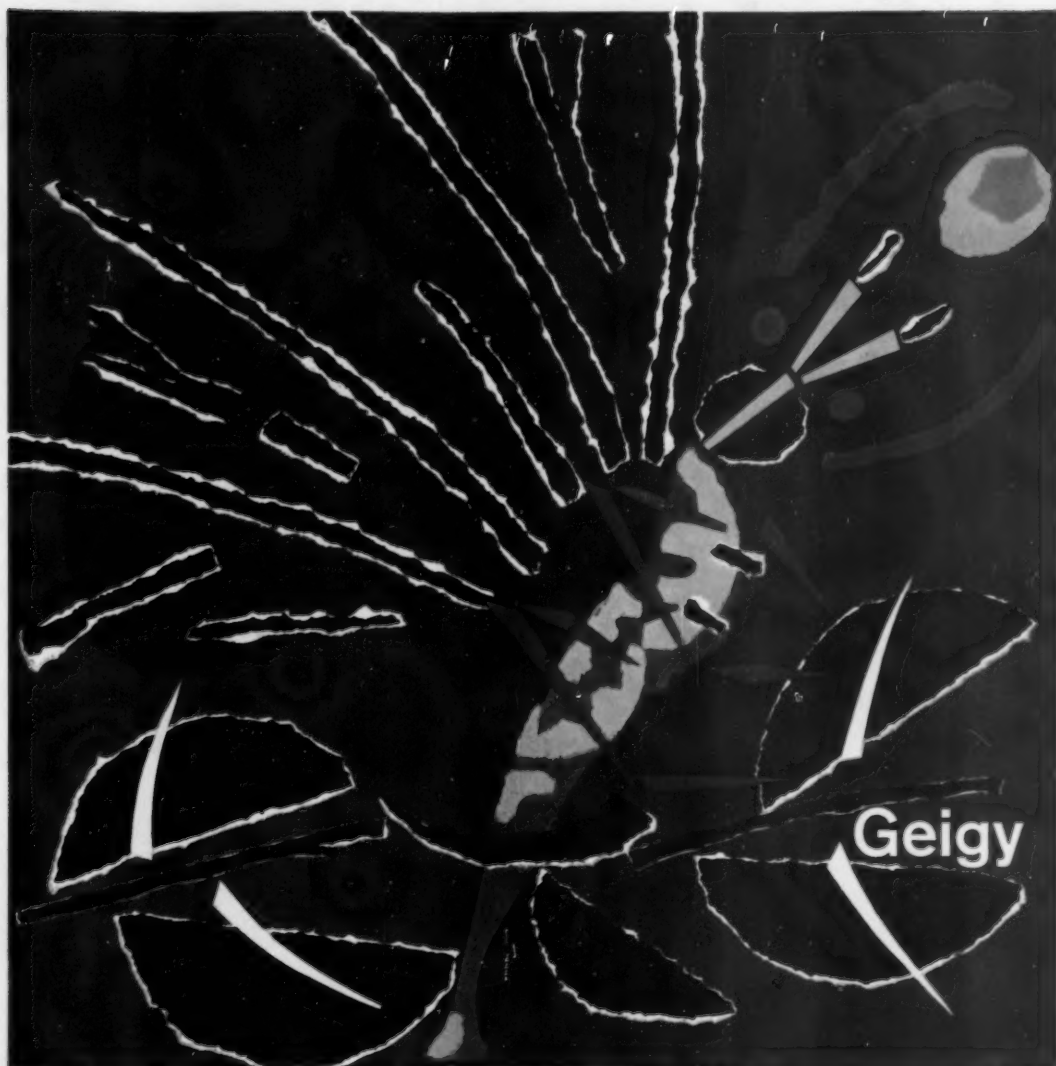
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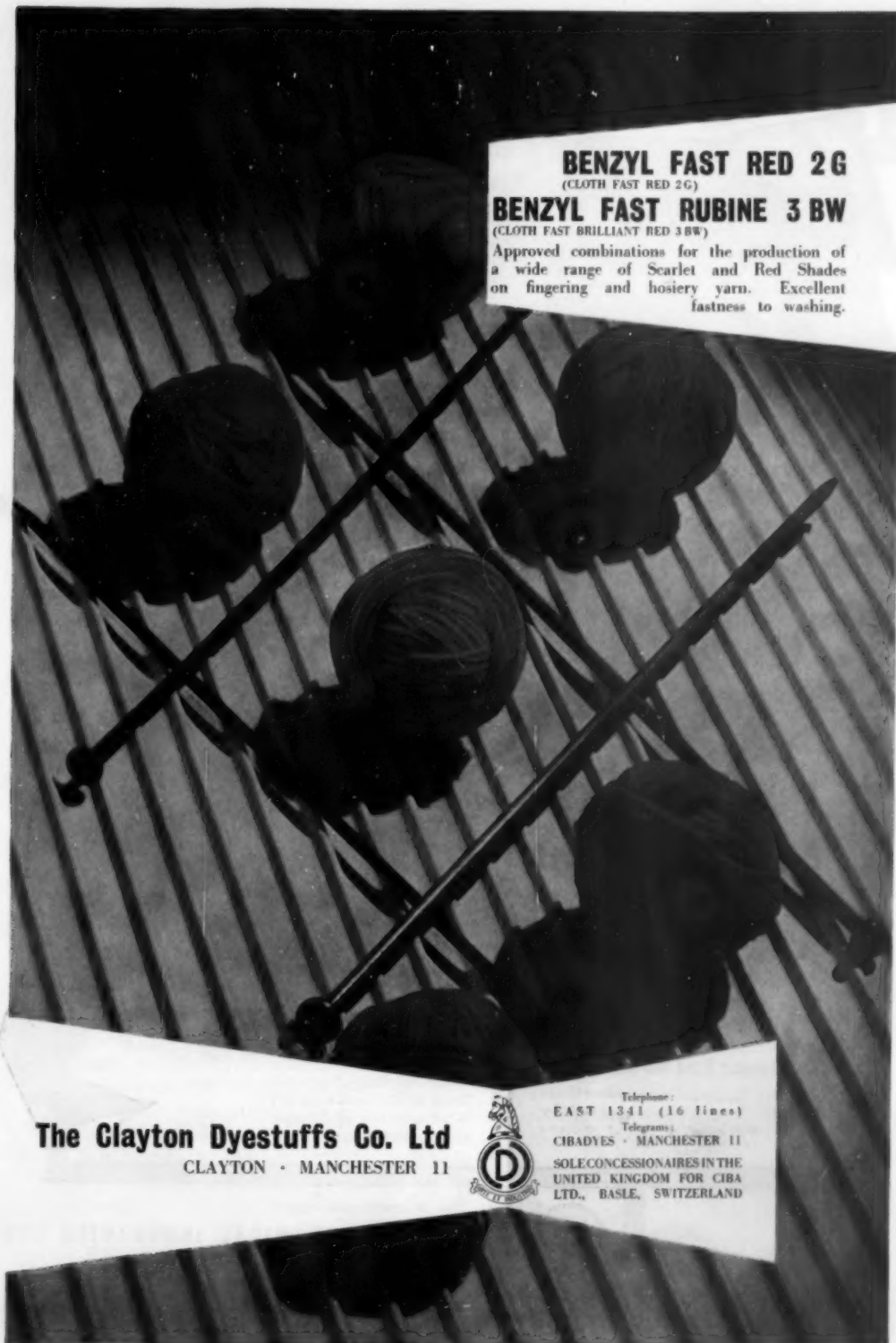
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Inns and the Textile Trade



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The 16th century market house was built by George Luttrell. Its lichen-covered timbers sheltered the traders and

their wares from the elements: its shape, octagonal rather like a dove-cote, gave good all-round sales-stands.

This picturesque yarn market is now empty; the Luttrell Arms has changed hands; the Dunster trade is dead. And the buying and selling of textiles is no longer centred round a fine old inn or market-house bequeathed by squire or lord. Bigger trade depends on cheaper, yet finer cloth; cheaper yet faster colours, more materials processed for special wear. This is largely the province of chemical research that seeks consistently to improve the cloths and give them qualities demanded by the modern public—important work that is conducted by companies like Brotherton.

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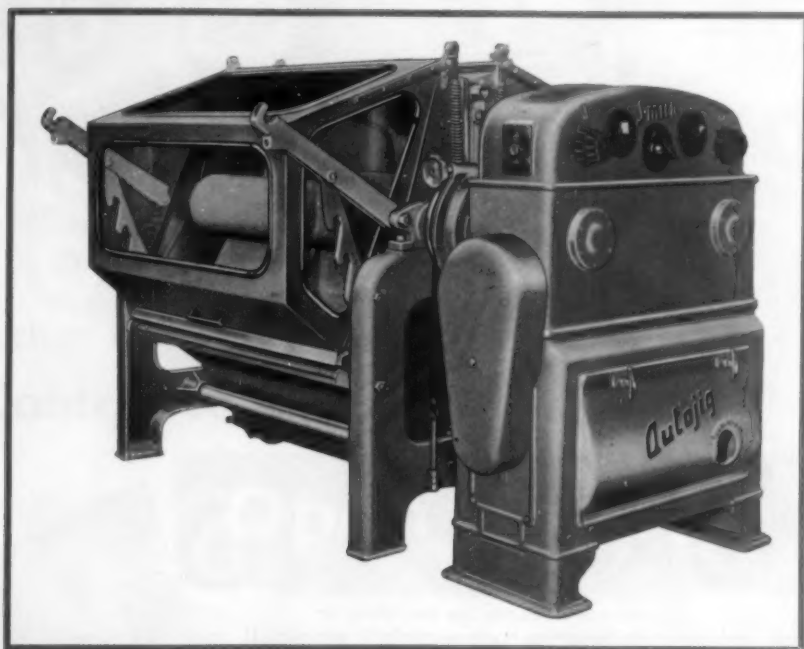
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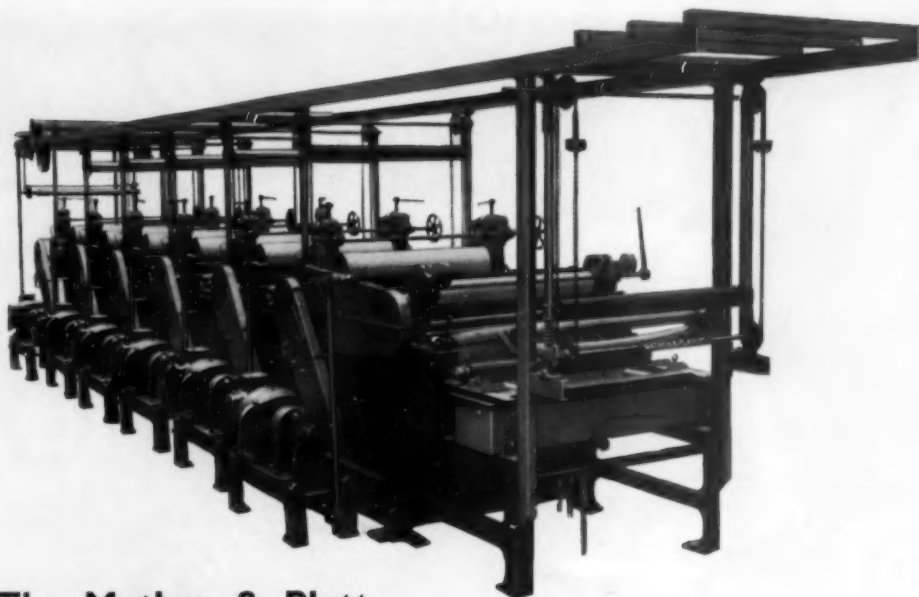
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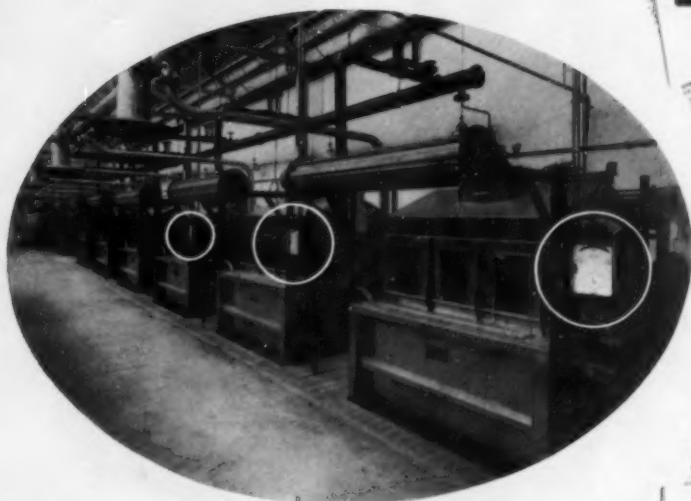
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and built specifically for textile finishing, and only one is laid out on one floor. Three of the plants are operated by independent commission finishers, one is a unit owned by a finishing combine, and the remaining three are either part of or closely allied to a vertical textile organisation.

Only two of the seven plants had any formal agreement with a trade union, and arrangements to accommodate changes in plant, processes, etc. as regards staffing and work loads are similar to current agreements in this country. It is notable that four of the plants studied had no piecework or similar incentive schemes, and in the other three only a proportion of the employees were paid by results.

Overall output per operative-day varied from 200 yd. at a small commission dyeworks to the phenomenal figure of 1,700 yd., the latter figure referring to two selected cloths only and not to the full output of the plant concerned. Daily output per operative at the remaining plants lies between 500 and 1,500 yd. Such a wide variation gives little basis for direct comparison with overall output in U.K. dyeworks. Details of output on individual processes may help to point problems for investigation, but the reader is told, at the commencement of the book, that these problems require the expert assistance of engineers, either within the plant or outside consultants.

H. MELLOR

Methoden der Organischen Chemie

(Houben-Weyl)

Band III

Physikalische Forschungsmethoden

Teil 2

Edited by Eugen Müller. 4th edition 1955. Pp. xxviii + 1078. Stuttgart: Georg Thieme Verlag. Price, DM 186.00.

This authoritative compilation on physical methods employed in organic chemistry is the second part of Volume III, and deals with electrical, optical, magnetic, and acoustical methods, in 21 chapters—(1) *Conductivity Measurements*, including general theoretical treatment and copious details of apparatus employed in the Wheatstone bridge and Pontavi-Wheatstone bridge methods, the special devices of Phillips and of Lange, and the dephimeter of Klutke-Draeger. (2) *pH Methods*, with comprehensive theoretical explanations and detailed laboratory determinations of pH values and redox potentials. (3) *Indicators and Reagent Papers*. (4) *Potentiometric and Conductimetric Titrations*. (5) *Quantitative Electrophoresis in a Separation Tube*, with relevant theory. (6) *Electrochemical Potential of Organic Compounds*, dealt with under potentials of systems in equilibrium and kinetics of the regulation of redox equilibria. (7) *Polarography* as applied to organic compounds. (8) *Methods for the Determination of Dielectric Properties*. (9) *Refractometry*. (10) *Polarimetry*, with fundamental theory and a description

of the production of linearly polarised light and of the polarimeter and saccharimeter. (11) *Dispersion of Light*. (12) *Fluorescence and Phosphorescence*, including a special section on the employment of fluorescent dyes. (13) *Electron Diffraction*. (14) *Electron Microscopy*. (15) *X-Ray Methods*. (16) *Absorption Spectroscopy in the Visible and Ultraviolet*, with 125 pages devoted to the relation between the chemical constitution and the absorption spectra of organic compounds. (17) *Raman*. (18) *Infrared*, and (19) *Microwave Spectroscopy*. (20) *Magnetochemical* and (21) *Supersonic Methods*.

The literature references are complete to the beginning, and also in some cases to the end, of 1954. Where large subjects like electron microscopy cannot be described in *extenso* owing to the great amount of space required, reference is made to suitable sources for further consultation. In some other chapters, e.g. (16), all existing material is treated as in a separate monograph, and, in the above section, about 250 spectra are included, which should be of inestimable value to the organic chemist. The largest chapter (18) (106 pages) deals with infrared spectroscopy, and should be invaluable for the determination of constitution, and for the identification and analysis of organic compounds.

It is impossible in any reasonable space to illustrate the high level of treatment of the themes discussed, but it gives pleasure to peruse this volume at leisure, since this confirms the impression of the great value of a work so well done. Some idea of its scope may be gained from the fact that the author index consists of 29 pages of names in three columns, and the subject index of 47 double-column pages—in fact it rivals that of Volume II on Analytical Methods. The diagrams (507), indexing, printing, and binding are excellent.

H. H. HODGSON

Color in Foods

A Symposium sponsored by the Quartermaster Food and Container Institute. Pp. vi + 186. Washington D.C.: National Academy of Sciences—National Research Council. 1954. No price.

This collection of papers is a good illustration of the extent to which quantitative colour measurement and standardisation can be used with apparent success in a highly psychological field. The individual contributions are in general short, and the presentation is attractive, gaining much from the verbatim style of the discussions.

The book will be of considerable interest to those concerned directly with the storage and marketing of food. For the general reader, much of the material is rather highly specialised, but he might read with advantage the three review papers included, and also be interested in the zeal with which photoelectric colorimeters and spectrophotometers are applied to colour problems in the U.S.A.

E. ATHEFTON

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index

I—PLANT; MACHINERY; BUILDINGS

Plastics Constructional Materials in Textile Fibre

Plants. W. A. Haldeman and E. F. Wesp. *Ind. Eng. Chem.*, 47, 1359-1366 (July 1955).

A review of the various uses to which plastics materials are put in the equipment employed in the production of man-made fibres. W. K. R.

Some Mechanical Improvements for Dyeing

Machines. H. Creogan. *Amer. Dyestuff Rep.*, 44, P 495-P 496 (18 July 1955).

Comments on Package-dyeing Machines. A. P. March. *Amer. Dyestuff Rep.*, 44, P 494, P 496 (18 July 1955).

Experimental Application of a Turbodynamic

Principle in Dyeing. D. Fornelli. *Amer. Dyestuff Rep.*, 44, P 456-P 458 (4 July 1955).

A description is given of an experimental dyeing machine (capacity 500-600 ml.) in which a revolving perforated circular cage rotates at 1000 r.p.m. in a circular box fitted with deflection plates, which force the internal liquor in a direction contrary to the rotation of the cage. This induces turbulence, and it is possible to dye wool below the boil (70°C.) at normal pressure. Under these conditions a felt was fully penetrated after 15 min. while a control was incompletely penetrated after 2½ hr. at the boil in a conventional dyepot. J. W. B.

Recent Developments in the Roller Printing Machine.

K. H. Bönkost. *Melliand Textilber.*, 36, 814-818 (Aug. 1955).

Innovations in drive, braking, frame, doctor, furnishing, mandrel, and registration are illustrated. S. R. C.

PATENTS

Thread Storing, Thread Advancing Reel. H. A. Kuljian. USP 2,694,308

In a reel of the type made up of rotating rolls a combined scraping or wiping and segregating device removes excess liquid in a segregated manner from the surface of the roll to which the liquid has been applied and from the surface of at least one adjacent roll. C. O. C.

Felting in the Bumping Stocks. British Hat & Allied Feltmakers Research Assoc. BP 735,583

A method for the better control of temperature and acid concentration. C. O. C.

Starch Cooking Apparatus. Springs Cotton Mills. BP 735,161

An apparatus for cooking starch comprises a container for slurry to be cooked and a separate cooking chamber. The slurry is circulated from the container to the cooker and back again, the return pipe having a terminal portion at least part of which extends vertically against the wall of and within the container. The cooked slurry is injected in such a way that a swirling motion is provided and hinders coagulation of the starch on the vat walls. Holes are provided above the level of the starch to ensure that entrained steam is released and does not cause boil-ups in the container. J. W. B.

Continuous Wet Processing of Freshly Wet-spun Man-made Fibres. American Cyanamid Co. BP 734,850

Continuous Wet Treatment of Freshly Formed Artificial Threads. H. A. Kuljian. BP 735,373

Embossing or Marking Press. Maason Seeley & Co. BP 734,502

A press in which ribbon, etc. is embossed and/or printed. C. O. C.

Hat Pouncing Machine. Doran Brothers. USP 2,694,513

Hosiery Form. Paramount Textile Machinery Co. BP 734,819

A hosiery form of oval cross section comprising two opposing metal plates flared away from each other along the rear longitudinal edge to accommodate the stocking seam. G. E. K.

Fabric Drier. H. J. F. Jauzac. BP 734,995

Fabrics are laid over an inverted U-shaped casing containing a gas-heated boiler supplying steam to a series of tubes spaced vertically inside, and in contact with the casing. G. E. K.

II—WATER AND EFFLUENTS

Treatment of Wool Scouring Wastes. W. H. Taylor. Sanitalk 1, 13-14 (Nov. 1952); Public Health Eng. Abstr., 33, (1-2) 17-18 (1953); Chem. Abs., 49, 9846 (25 July 1955).

Treatment of the waste with CaCl_2 and then passing through a Sharples DG-2 centrifuge results in reduction of the fats, turbidity and suspended solids by 98-99%. Best results are got by adding the CaCl_2 soln. 3 sec. before centrifuging and then holding the effluent in a skimming tank for 20-30 min. Temperature had little effect. C. O. C.

Characteristics of Wool Dyeing Wastes and Experimental Treatment. J. A. McCarthy. Sanitalk 1, 8-11 (Nov. 1952); Public Health Eng. Abstr., 33, (1-2), 17 (1953); Chem. Abs., 49, 9846 (25 July 1955).

Waste from top dyeing contains little suspended matter and much of the biological oxygen demand (B.O.D.) is in the solution. Ordinary coagulants are generally ineffective and even prolonged sedimentation gives little improvement. Storage in lagoons may result in some reduction in colour but little reduction in B.O.D. With standard trickling filters loadings of 1000-1500 lb. of B.O.D. per acre foot resulted in > 90% B.O.D. reduction. Recirculating the waste through the filters with a ratio of 5:1 showed that B.O.D. loading could be gradually increased to 5000 lb. per acre foot with an 89% B.O.D. reduction. Vat dyeing waste, though lower in B.O.D., required a reduction in filter loading to 1000 lb. B.O.D. per acre foot to obtain a 90% reduction. Recirculation of this waste through the filters increased the loading by only 30-40%. As both these types of dye wastes are customarily found in the same mill (in Massachusetts), combining the two wastes gave satisfactory effluent through trickling filters with loadings of 1200 lb. B.O.D. per acre foot. C. O. C.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Polyphosphates in Textile Processing. K. Schuster. Textil Praxis, 10, 600-604 (June 1955).

Polyphosphates and their use in hard water and water containing metals are detailed. In addition, their applications in various branches of textile processing, including desizing, kier boiling, bleaching, scouring, dyeing, printing, and milling, are outlined. B. K.

Characteristic Properties of Polyethylene Oxide

Products. C. Schöller. *Melliand Textilber.*, 36, 603-607 (June 1955).

The solubilities, foaming powers, stabilities in storage, influences of raw materials and molecular structure, wetting powers, and effects of electrolytes are referred to. S. R. C.

Complexones. XXVI—Coordinating Power of N-

Substituted Iminodiacetic Acids. G. Schwarzenbach, G. Anderegg, W. Schneider, and H. Senn. *Helv. Chim. Acta*, 38, 1147-1170 (Aug. 1955).

Substituted iminodiacetic acids of the general formula $\text{R-N}(\text{CH}_2\text{-COOH})_2$ have been synthesised, and their coordinating powers with 12 different metal cations investigated. Thirteen different radicals R were employed and aliphatically bound O, S, and N formed a third chelate ring in the iminodiacetic anion-metal cpd. When R was $-\text{CH}_2\text{-CH}_2\text{-S}-$ a particularly stable complex was formed. H. E. N.

Annual Review of the Literature on Fats, Oils, and

Detergents. M. Piskur. *J. Amer. Oil Chem. Soc.*, 32, 265-271 (May); 319-341 (June 1955).

P. G. M.

Solid Non-ionic Surface-active Agents. G. E. Barker and H. J. Ranauts. *J. Amer. Oil Chem. Soc.*, **32**, 249-252 (May 1955).

Methods have been described for converting liquid non-ionic surface-active agents of the polyoxyethylene type to solid compositions by complexing with urea. It is shown how these solid products may be made in the form of free-flowing powders, flakes, bars, pellets or tablets.

The surface-active properties of two commercial detergents based on this discovery are reported. Besides showing the excellent detergency of these products in washing artificially soiled cotton, it has been shown that they enhance the detergency of alkali-built sodium alkylarylsulphonates in hard water.

P. G. M.

PATENTS

Detergent for Use in Salt or Hard Water. H. F. Johnston. *BP* 734,982

A mixture of commercial stearic acid, a sulphonated fatty oil, Na or K silicate, NaOH and/or K_2CO_3 , a non-ionic detergent and water, with or without a sulphated fatty alcohol, the fatty acid content being 4-19%, is an efficient detergent in salt or hard water.

BP 735,418

The mixture includes a secondary alkyl sulphate.

C. O. C.

Detergent. Colgate-Palmolive Co. *USP* 2,692,235

Addition to synthetic detergents, particularly anionic sulphated or sulphonated detergents, of a small proportion of a compound of formula R^1-X-SO_2-NYZ (R^1 = Alk of 10-22 C; X = mononuclear aromatic radical; Y and Z = same or different, H or Alk of 1-5 C) e.g. dodecyl benzene sulphonamide, achieves a significant and synergistic enhancement in detergency.

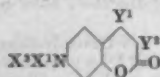
USP 2,692,237

Compounds of formula R^1-SO_2-NYZ (R^1 = Alk of 8-18 C), e.g. dodecyl or lauryl sulphonamide, may be similarly used.

C. O. C.

Fluorescent Brightening Agents for Use in Washing Nylon Goods. Pro-Nyl Chemicals. *USP* 2,692,238

3:7-Dianisoylamidodibenzothiophenesulphonic acid and compounds of formula—



(X^1 and X^2 = same or different Alk; Y^1 and Y^2 = same or different, H or Alk), e.g. *p*-diethylaminocoumarin (Blanco-phor AW (G)), can be used in detergent solutions. They are useful for making up into concentrated detergent or rinsing mixtures, e.g. an aqueous soln. containing 0.1-0.6% mineral acid, and 20% of a monohydric alcohol having dispersed in it 0.1-6.0% of an aminocoumarin is added to the rinse water for nylon articles.

C. O. C.

Organophilic Silica Organosols. DuP.

USP 2,692,863

Addition of a long-hydrocarbon-chain-substituted ammonium base, e.g. cetyl trimethyl ammonium bromide, simultaneously with or before addition of a brine-immiscible organic liquid, e.g. butanol, to an aqueous silica sol. of particle size 10-150 μ , results in the silica being transferred to the organic liquid. The resulting organosols have a wide variety of uses including those of emulsifying and wetting, delustring, anti-slip and stiffening agents for textiles, etc.

C. O. C.

Warp Size. Atlas Powder Co. *USP* 2,694,021

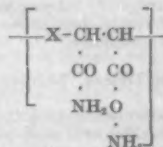
An efficient size for rayon warps intended for fabrics having a large number of ends per inch consists of an aqueous solution containing 5-15% of starch or acid-modified starch and 0.05-2.0% of polyoxyalkylene higher fatty acid, e.g. 8'-hydroxypolyethoxyethyl stearate.

C. O. C.

Water-soluble Nitrogenous Copolymers. Monsanto.

BP 735,263

The products obtained by treating polymers of maleic acid and vinyl or isopropenyl acetate with anhydrous NH_3 are polyelectrolytes, i.e. they are high mol. wt. polymers which ionise in aqueous solution. They are made up of—



(X = residue of the acetate monomer) units. They are useful for application to textiles or paper and as stabilisers for aqueous emulsions or dispersions.

C. O. C.

Antistatic Agent. DuP.

USP 2,694,688

An aqueous dispersion containing a water soluble polymer of a quaternary salt of an acryloyloxyalkyltri-alkylamine (each Alk of 1-4 C) (15 parts by wt.), e.g. poly- β -methacryloyloxyethyl-diethylmethylammonium methyl sulphate, a water-soluble salt of an aliphatic alcohol sulphate of 8-20 C (2-10), e.g. sodium hexadecanoyl sulphate, a satd. aliphatic monohydric alcohol of 2-12 C (5-10), e.g. ethyl or isopropyl alcohol, mixed monocyclic terpene hydrocarbons (2-20), and water (to bring to 100), is an excellent antistatic agent for acetate rayon, nylon, Terylene, etc., the effect being resistant to washing.

C. O. C.

Flame-resisting Agent. National Lead Co.

USP 2,691,594

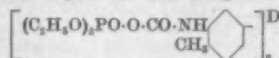
A solution for rendering cellulosic materials flame-resisting consists of an aqueous solution of hydrochloric acid and titanium chloride formate, acetate or propionate, the amounts of HCl and the titanium chloride acylate being such that there is 90-200 g. of chlorine per litre. The solution is applied at $> 60^\circ\text{C}$. and is dried into the material in the normal manner. The flame-resisting finish obtained is fast to washing.

C. O. C.

Flame-resisting Finishing or Anti-static Agents. DuP.

USP 2,691,566

Compounds containing a phosphate, phosphonate, phosphinate or phosphine oxide radical and the group $-R^1-D-R^2-$ (R^1 and R^2 = aryl radicals; D = addition product of two isocyanate radicals), e.g. the compound—



formed by treating diethyl phosphoric acid with the dimer of 2:4-tolylene diisocyanate, are excellent flame-resisting agents for cotton. Applied from aqueous dispersion and baked at 180-190°C. for 3 min. they give a finish which is fast to washing and dry cleaning. Applied to nylon in the same way, there results an anti-static finish which is fast to repeated washings.

USP 2,691,567

Derivatives of starch, cellulose, polyvinyl alcohol or polyallyl alcohol containing aromatic diisocyanate dimers attached by means of urethane linkages and some of the hydroxyl groups substituted by substituted phosphate, phosphonate or phosphinate radicals, e.g. the polymer obtained by partly esterifying polyvinyl alcohol with diethyl phosphoric acid and then treating it with the dimer of 2:4-tolylene diisocyanate are applied to fabrics from solvents or emulsions, dried and baked at 125-200°C. Applied to cellulose, cellulose acetate, nylon and Terylene, so as to add 2.5-3.0% phosphorus, they yield an excellent flame-resisting finish which is fast to dry cleaning and washing; applied in very much smaller amounts (0.05% by wt.) they impart an anti-static finish.

C. O. C.

Flame-resistant and Softening Composition for Cellulosic Materials. Heyden Chemical Corp.

USP 2,692,203

A flame-resistant and softening composition for treating paper, cellulosic textiles, etc., consists of the product obtained by reacting together a dibasic acid amide, e.g. urea, a hygroscopic polyhydric alcohol, e.g. glycerine, and an "oxide of a phosphorus compound containing hydrogen", e.g. diammonium phosphate or phosphoric acid.

C. O. C.

Solvent for Acrylonitrile Polymers. BrC.

BP 734,527

Polymers containing $< 85\%$ of acrylonitrile readily dissolve at 70-80°C. in mixtures of 30-90% of ethylene

carbonate and 70–10% of 1:2-propylene carbonate to yield solutions which can be kept at room temperature for 1–3 days before they solidify. C. O. C.

Metal-free Organic Driers. Sherwin-Williams Co. USP 2,694,015

Aliphatic aldehydes of 7–18 fC, e.g. octadecenyl aldehyde, 2-ethylhexaldehyde, *n*-decylaldehyde, 3:5:5-trimethyl hexaldehyde, citronellal and cinnamic aldehyde, are effective driers for drying oils, 0.5–1.75% on the total solids content of the composition giving the best results. C. O. C.

Imparting Permanent Fragrance to Fabrics. H. Pichlmayr. BP 735,684

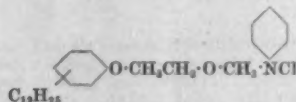
An etheral oil is dissolved in the plasticiser used for an aqueous dispersion of a synthetic resin used as a permanent stiffening agent for textile articles. C. O. C.

Resin Finish to Impart Water-repellency, Dimensional Stability and Recovery from Creasing. Sun Chemical Corp. USP 2,693,460

Urea, thiourea, mono-alkylated urea, mono-alkylated thiourea, guanyl urea, biuret, dicyandiamide or guanidine, is condensed with formaldehyde and with a lower aliphatic alcohol first under alkaline and then under acid conditions followed by reacting the condensate with a hydrophobic amide, amine or alcohol so as to obtain a product which on heating becomes insoluble. The products are used to render textiles water-repellent, dimensionally stable and able to recover from being creased. C. O. C.

Water-repellent Finish. General Aniline. USP 2,693,430

Compounds of formula $Y-Ar-O-(CH_2-CH(R^1)R^2-O)_n-CH_2-N$ (tert.)-Hal (Y = nuclear alkyl group of 7 C; $n = 1-3$; R^1 and $R^2 = H$ or Alk; $n =$ a whole number; Ar must be free from water-solubilising groups and/or reactive substituents) are water-soluble cationic surface active agents yielding colloidal solutions in water and having outstanding dispersing, wetting and emulsifying properties. They are used to render textiles water-repellent. Thus—



applied as a 2–5% aqueous solution to cellulosic fabric which is then dried and baked at 120–160°C. for 10–3 min. imparts an excellent water-repellent finish which is fast to dry cleaning and laundering. C. O. C.

Water-repellent Finishing Agents. General Aniline. USP 2,692,837

Methylolamides of higher aliphatic carboxylic acids (e.g. those corresponding to the natural fatty glycerides) when combined with compounds of formula $A-CH_2-N$ (tert.)-X (A = alkyl carbonamide of 8–18 C, alkoxy of 8–18 C, alkoxy mono- or polyethenoxy ether radical (alkoxy of 8–18 C) containing 1–3 ethenoxy groups, fatty acid esters of hydroxy mono- or polyethenoxy ether radicals (fatty acid of 8–18 C) and containing 1–3 ethenoxy groups, or nuclear alkyl-subst. phenoxy mono- and polyethenoxy ether radical (nuclear Alk together contain 5–18 C); N-tert. = radical of a tertiary N base; X = anion of a volatile acid) readily form aqueous emulsions. Textiles impregnated with such emulsions, dried and baked, are given a water-repellent finish which is fast to washing and dry cleaning without any yellowing of light-coloured fabrics. A suitable composition consists of methylol stearamide (1–2 parts), stearylamine methyl pyridinium sulphite (1) and isopropyl alcohol (2–3). C. O. C.

Antifungal Agent from Osage Orange Wood: Dyes present in the Wood (IV p. 676).

Paper Chromatography of the Non-tannins of Tanning Extracts (XII p. 693).

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Electronic Spectra, Hyperconjugation, and the Baker-Nathan Effect. A. Burawoy and E. Spinner. J.C.S., 2085–2088 (July 1955).

The spectra (*K*-bands) of diphenyl, acetophenone and its oxonium salt formed in conc. H_2SO_4 , aniline, the phenoxide ion, and some of their 4-alkyl deriv. indicate that the electron-donating character of the alkyl groups increases in the normal inductive order $CH_3 < C_2H_5 < (CH_2)_3CH < (CH_2)_4CH_3$, and that the differences between the alkyl groups are enhanced with the increase of electron demand. The absence in electronic spectra of a hyperconjugative electron-release increasing in the order $(CH_2)_3C \rightarrow CH_3$ is discussed, and the Baker-Nathan effect is ascribed to a steric resistance to bond contraction and stabilisation, these being reduced with the increasing size of the alkyl group. This interpretation, in contrast to that based on hyperconjugation, involves only inductive electron displacements. H. H. H.

Acyl-1- and -2-naphthols. N. P. Buu-Hoi and D. Lavit. J. Org. Chem., 20, 823–828 (July 1955).

Authentic 4-lauroyl-, 4-palmitoyl-, and 4-stearoyl-1-naphthol are prepared by demethylation of the corresponding 1-methoxynaphthalene and are shown to differ from the cpd. recorded in the literature under these names. The 4-*n*-decanoyl-, *n*-caproyl-, hexahydrobenzoyl-, myristinoyl-, and phenacetoyl deriv. are synthesised. Authentic 1-stearoyl-2-naphthol also differs in properties from the cpd. recorded in the literature. H. E. N.

Reaction of some Olefin Oxides with Naphthols. C. O. Guss, R. Rosenthal, and R. F. Brown. J. Org. Chem., 20, 909–919 (July 1955).

Mechanism of Formation of Diphenyl from Fluorobenzene and Phenyl-lithium. E. F. Jenney and J. D. Roberts. Helv. Chim. Acta, 38, 1248–1254 (Aug. 1955).

Using 1- ^{14}C -fluorobenzene, it is shown that the radioactivity in the diphenyl is distributed equally between positions 1 and 2. A symmetrical intermediate, probably benzyne C_6H_4 , must therefore have been formed. H. E. N.

Preparation of 4:5-Benzointhane, 1-Methyl-4:5-benzointhane, and 2:3-cycloPentenoacenaphthene. H. Dannenberg and A.-U. Rahman. Chem. Ber., 88, 1405–1415 (Sept. 1955).

Addition of Maleic Anhydride to Styrene [Naphthalenepolycarboxylic Acids]. K. Alder and R. Schmitz-Josten. Annalen, 595, 1–37 (Sept. 1955).

On refluxing in benzene, in the presence of hydroquinone and iodine as polymerisation inhibitors, naphthalene-1:2-dicarboxylic acid and its tetrahydro deriv. are produced. In presence of picric acid as inhibitor two adducts of 2 mol. of maleic anhydride and 1 mol. of styrene are formed. The one obtained in much greater amount is converted by dehydrogenation (Br_2 -acetic acid) and oxidation ($KMnO_4$) into naphthalene-1:2:4-tricarboxylic acid, synthesised from α -methylstyrene and maleic anhydride, via 1-methyl-naphthalene-3:4-dicarboxylic acid. The other product, derivatives of both, and the mechanism of the reaction are described and discussed. H. E. N.

Substitution-Addition of Maleic Anhydride and Hydronaphthalenes and Fluorene. K. Alder, H. Wollweber, and W. Spanke. Annalen, 595, 38–54 (Sept. 1955).

1:3:3:6-Tetramethyl-1-*p*-tolylindane. A. V. Grampoloff. Helv. Chim. Acta, 38, 1263–1268 (Aug. 1955).

When *p*-cymene is alkylated with *tert*.-butyl or *tert*.-amyl alcohol in presence of sulphuric acid, alkyl-*p*-cymenes are not formed, but amongst the alkylated indanes obtained the above cpd. is found. H. E. N.

Allegedly Isomeric Diazo Derivatives from "Dehydrothioparatoluidine" (2-*p*-Aminophenyl-6-methylbenzothiazole). M. Aroney and R. J. W. Le Fèvre. J.C.S., 2138–2142 (July 1955).

Morgan and Webster's 5-methyl-1-phenylbenzothiazole-4'-*syn*- and -*anti*-diazochlorides (J.C.S., 119, 1072 (1921))

are shown to be respectively the diazonium chloride from, and the monohydrochloride of, 2-*p*-aminophenyl-6-methylbenzothiazole. Certain sparingly sol. "diao deriv." of the alleged "syn-chloride" should be viewed as diazonium salts with complex anions. Evidence is given that Morgan and Webster prepared an authentic diazocyanide. As yet no covalent diazochloride has been isolated, although such structures may be transient intermediates in certain reactions (cf. Hodgson, *J.S.D.C.*, 58, 228 (1942)). H. H. H.

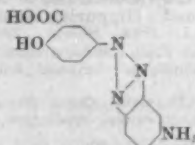
Reduction of Cytotoxic Azo Compounds by Hydrazine and by the Xanthine Oxidase-Xanthine System. W. C. J. Ross and G. P. Warwick. *Nature*, 176, 298-299 (13 Aug. 1955).

Aromatic Diazo Compounds. XVII—Dark Brown Direct Cotton Dyes of Superior Light Fastness. J. Poskocil and Z. J. Allan. *Chem. Listy*, 49, 1782-6 (1954); *Chem. Abs.*, 49, 7855 (10 June 1955).

Direct cotton browns of the type of Sirius Brown RT were found to be improved in light fastness by coppering or by removal of NH_2 groups by diazotisation and reduction with sodium formate or acetate. Thus, the trisazo dye salicylic acid \leftarrow benzidine \rightarrow 1-naphthylamine-6-sulphonic acid \rightarrow 1-naphthylamine-6-sulphonic acid was diazotised at 10-12°C. and the solution so formed heated for 1 hr. at 60-65°C. with sodium formate or acetate followed by salting out.

XVIII—Red and Violet Direct Cotton Dyes Derived from Benzotriazole. F. Muzik and Z. J. Allan. *Ibid.*, 1787-1793; *Chem. Abs.*, 49, 7856-7857 (10 June 1955).

The monoazo dye 5-aminosalicylic acid \rightarrow *m*-phenylenediamine, dissolved in aq. NaOH and oxidised with air in presence of MnSO_4 and CuSO_4 , gave 2-(4'-hydroxy-3'-carboxyphenyl)-5-aminobenzotriazole—



This compound and its derivatives were diazotised and coupled with suitable coupling components to give dyes; e.g. urea-di-J-acid gives a bright red. E. S.

Subsidiary Dyes in FD&C Colours. Lower Sulphonated Dye in FD&C Blue No. 1. M. Dolinsky. *J. Assoc. Offic. Agr. Chemists*, 38, 359-365 (1955); *Chem. Abs.*, 49, 9931 (25 July 1955).

Several low sulphonated derivatives of FD&C Blue No. 1 (Brilliant Blue FCF) can be obtained by condensing 1 mol. of benzaldehyde or a benzaldehyde sulphonic acid with a mixture of a sulphonated and an unsulphonated amine. The resulting leuco compounds are oxidised and the dyes separated by solvent extraction. The subsidiary dyes found in commercial samples of FD&C Blue No. 1 are disulphonated and appear to be a mixture one component of which is the dye obtained by substituting *N*-ethylaniline for sulphonated *N*-ethyl-*N*-phenylbenzylamine and which seems to be a decomposition product, possibly formed when alkali is added during the manufacture of FD&C Blue No. 1. C. O. C.

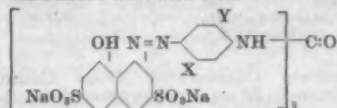
Copper Complexes of Some Direct Cotton Azo Dyes. V. Krepelka and J. Rais. *Rozprawy české akad.*, 61, No. 3, 1-19 (1951); *Bull. intern. acad. technique*, 52, 543-4 (1951); *Chem. Abs.*, 49, 9929 (25 July 1955).

The substantivity and tinctorial power of some sym. diazo and the parent monoazo dyes, and their Cu complexes, derived from *o*-aminophenol, *o*-aminosalicylic acid, 3:3'-diamino-4:4'-dihydroxydiphenylmethane, 5:5'-methylenebis(3-aminosalicylic acid), bis-*p*-aminophenylurea, benzidine-3:3'-disulphonic acid, benzidine sulphone, 3:3'-disulphobenzidine sulphone, and diamino stilbene-2:2'-disulphonic acid, were examined. Acid conditions of metalisation, using saturated CuSO_4 , gave less soluble complexes of lower tinctorial power and higher substantivity, whereas the use of ammoniacal CuSO_4 brought about smaller changes in these properties. Dyes lacking sulpho groups bound 1 atom of Cu per 2 azo groups under

acid conditions, and 1 atom of Cu per 1 azo group under alkaline conditions; dyes containing sulpho groups did not bind Cu according to any definite rule. Dyes exhibiting an increased ability to form complexes (e.g. COOH and OH groups in *ortho* position) were characterised by increased fastness and lower solubility of their Cu complexes. E. S.

Azo Dyes and their Intermediates. XIII—Synthesis of Chlorantine Fast Red 6BL. T. Nishi, K. Takagi, and Y. Abe. *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 57, 649-650 (1954); **XIV—Synthesis of Dyes in the Chlorantine Fast Red 6BL Series.** *Ibid.*, 651-2; *Chem. Abs.*, 49, 8601-3 (25 June 1955).

XIII—Chlorantine Fast Red 6BL—



(X = CH_3 ; Y = CH_3CO) was made in the following ways: (1) H-acid was esterified with *p*-toluenesulphonyl chloride and NaOH at 65-85°C. for 180-270 min. to give a 52-88% yield; (2) diazotised H acid ester dispersed in water was treated with 5-methyl-*o*-anisidine in presence of HCl and Na acetate at 5-10°C. for 3 hr. and then at 40°C. for 30 min. to give a 92% yield of 1-(4-amino-5-methoxy-2-methylphenylazo)-8-(*p*-methylphenylsulphonyloxy)-3:6-naphthalene sulphonic acid, this was treated with COCl_2 at 40-45°C. for 3 hr. in an aq. soln. of Na acetate and Na_2CO_3 , the product was then boiled in NaOH for 30 min., neutralised with acetic acid and salted out with Na acetate.

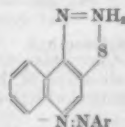
XIV—Dyes of the above formula where (1) X = Y = H; (2) X = H; Y = CH_3 ; (3) X = CH_3 , Y = H, and (4) X = H, Y = CH_3CO have been prepared by procedures similar to the above. They are red to dark red dyes of good light fastness on rayon or silk and satisfactory fastness on cotton. Their fastness to washing is good. CH_3 in the *ortho* position and CH_3CO in *meta* position exert a pronounced darkening effect. C. O. C.

Macrocyclic Ring Systems. II—Bis-(*NN'*-alkylene-benzidines). H. Stetter and E.-E. Roos. *Chem. Ber.*, 88, 1390-1395 (Sept. 1955).

NN'-Ditosylbenzidine is successfully condensed with ethylene dibromide, 1:3-dibromopropane, and 1:4-dibromobutane, and the products are hydrolysed. Bis-(*NN'*-tri- and tetra-methylenebenzidine) form clathrate cpd. with solvents, but the dimethylene deriv. does not, as is expected from models. H. E. N.

Azo Derivatives of 2-Amino- β -naphthothiazole. R. Passerini and L. Pentimalli. *Boll. sci. fac. chim. ind. Bologna*, 12, 110-3 (1954); *Chem. Abs.*, 49, 7855 (10 June 1955).

Dyes of formula—



(Ar = 2-thiazolyl, phenyl, *p*-nitrophenyl, 2:4-dinitrophenyl, *p*-bromophenyl, 2-benzothiazolyl, 6-methoxy-2-benzothiazolyl and 6-bromo-2-benzothiazolyl) have better affinity for and are faster on nylon than acetate rayon. The fastness of the dyeings to light, perspiration and washing are tabulated. Introducing a second thiazole nucleus did not cause any essential alteration in properties of the dyes. C. O. C.

Indazole Derivatives—Synthesis of Various Amino- and Hydroxy-indazoles and Derived Sulphonic Acids. R. R. Davies. *J.C.S.*, 2412-2423 (July 1955).

Indazoles prepared from various substituted *o*-toluidines are described, and evidence is given that 3-nitro-2:6-xylidine affords a mixture of 6- (36%) and 4-nitro-7-methylindazole (64%). Chlorination of 6-nitroindazole gives 3-chloro-6-nitroindazole, whereas sulphonation and nitration give the 5-substituted deriv. The preparation of several new amino- and hydroxy-indazolesulphonic acids is described, and tentative structures are assigned to the products. H. H. H.

Crystal Structure of Methylene Blue. J. O. Warwicker. *J.C.S.*, 2531 (July 1955).

X-Ray examination of Methylene Blue showed that the unit cell is monoclinic— $a = 9.63 \pm 0.02$, $b = 31.1 \pm 0.1$, $c = 6.96 \pm 0.02$ Å; $\beta = 97^\circ$ —and the space group is $P2_1/c$ (cf. similar results by Taylor, *Z. Krist.*, **91**, 450 (1935); *Chem. and Ind.*, 732 (1935)). Assuming the unit cell to contain four dye mol., the cell const. and the measured density of the crystals (1.21 g./c.c.) lead to a mol. wt. of 408, indicating a pentahydrate. Within the range 0–50% R.H. the moisture content of the dye increases continuously with increasing humidity, rising abruptly between 50 and 60% R.H. and remaining constant above 60% R.H., indicating the stable form of the dye to be a pentahydrate. No evidence of the existence of a tetra- or lower hydrate has been obtained.

H. H. H.

Coordinatively Unsaturated Nickel Complex of *N*-Phenyl-*N'*-*o*-carboxyphenyl-*C*-2-quinoxalinyformazan. M. Seyhan. *Chem. Ber.*, **88**, 1454–1455 (Sept. 1955).

Reactions with Formamide. VI—Conversion of Oxazoles into Iminazoles. H. Brederick, R. Gompper, and H. Wild. *Chem. Ber.*, **88**, 1351–1352 (Sept. 1955).

Certain oxazoles substituted by alkyl or aryl in positions 2, 4, and 5 cannot be converted into iminazoles by boiling with formamide, but they are now successfully treated with formamide and ammonia at 200–210°C. in an autoclave.

H. E. N.

Pigments from 3:3'-Dichloro- and 3:3'-Dimethoxy-4:4'-diaminostilbene. D. W. Hein and E. S. Pierce. *J. Amer. Chem. Soc.*, **77**, 4107–4110 (5 Aug. 1955).

A range of pigments has been prepared by tetrazotising 3:3'-dichloro- and 3:3'-dimethoxy-4:4'-diaminostilbene and coupling with variously substituted acetanilides, 1-phenyl-3-methyl-5-pyrazolone and 3-hydroxy-2-naphthoic acid arylides.

These pigments show the expected bathochromic colour shifts when compared with the corresponding pigments from 3:3'-dichlorobenzidine but valid comparisons can only be made among these compounds when more accurate data have been obtained.

C. O. C.

Triarylmethyl Radicals. I—Tri-*o*-tolylmethyl. W. Theilacker and M.-L. Wessel-Ewald. *Annalen*, **594**, 214–224 (Aug. 1955).

Hexa-*o*-tolylethane is much more dissociated than the corresponding *meta* and *para* compounds and hexaphenylethane, yet resonance within the radicals must be more difficult because of their non-planarity. Steric effects in the ethane must be important. The absorption spectrum of tri-*o*-tolylmethyl exhibits only an inflection where triphenylmethyl has its characteristic "radical absorption band". Semi-empirical calculations are performed on possible configurations of tri-*o*-tolylmethyl.

II—Tri-(*o*-ethylphenyl)methyl. W. Theilacker, B. Jung, and W. Rohde. *Ibid.*, 225–228.

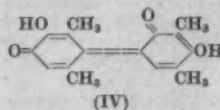
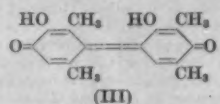
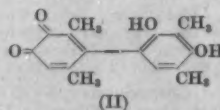
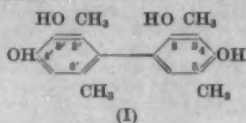
The extent of dissociation of hexa-(*o*-ethylphenyl)ethane is practically identical with that of tri-*o*-tolylethane.

H. E. N.

Red Colour given by Coal-tar Phenols and Aqueous Alkalies. Isolation of a Quinone derived from a Tetrahydroxytetramethyldiphenyl. W. Baker and D. Miles. *J.C.S.*, 2089–2096 (July 1955).

The red colours referred to in the title are due to simultaneous aerial oxidation of *C*-alkylated catechols and resorcinols to form compounds of the type afforded by oxidative coupling of 3:5-dimethylcatechol and 2:4-dimethylresorcinol. The latter reaction, limited by nuclear substitution, affords, almost quantitatively, the crystal, mono-Na salt of a quinone derived from 2:4:3':4'-tetrahydroxy-3:5:2':6'-tetramethyldiphenyl (I), which is shown to be a tautomeric substance capable of reacting in the *o*-benzoquinonoid form (II) and one or both of the diphenquinone forms (III) and (IV). The effects of steric factors on the central C-C bond in these quinones are discussed. Although the salt of this quinone is not the only colouring matter formed by the alkaline oxidation of phenols from low-temp. carbonisation and other coal tars, it seems likely, from the present work, that the compounds which contribute to the more purely red

colours are quinones derived from 2:4:3':4'-tetrahydroxydiphenyl, among which the above salt may well be of major importance.



H. H. H.

Reaction between Rhodamine B and Antimony. R. W. Ramette. *Univ. Microfilms* (Ann Arbor, Mich.), Publ. No. 11,116, 122 pp.; *Dissertation Abs.*, **15**, 348–9 (1955).

Fluorescence of the Condensates of Some Aromatic Aldehydes and Hippuric Acid (Azlactones). J. Eisenbrand. *Arch. Pharm.*, **296**, 441–7 (1953); *Chem. Abs.*, **49**, 7555 (10 June 1954).

Several compounds of formula 4:3-R'O(R'O')C₆H₃·CH₂:N:CC₆H₄:O·CO show green to greenish yellow fluorescence under ultraviolet irradiation.

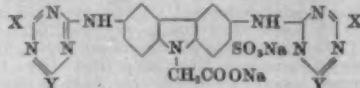
C. O. C.

Syntheses of Organic Fluorescent Compounds.

XXVI—Syntheses of 9-Carboxymethyl-3:6-diamino-1:8-carbazoledisulphonic Acid Derivatives with Different Substituents. R. Oda, Z. Yoshida, and R. Yasuda. *J. Chem. Soc. Japan, Ind. Chem. Sect.*, **56**, 549–551 (1953); XXVII—Synthesis of 9-Carboxymethyl-3:6-bis(4-hydroxy-6-substituted-2-*s*-triazylamino) carbazolemonosulphonic Acid. Z. Yoshida and R. Yasuda. *Ibid.*, **711–3**; XXVIII—Synthesis of 9-Carboxymethyl-3:6-bis(4:6-disubstituted-2-*s*-triazylamino) carbazolemonosulphonic Acid. *Ibid.*, **713–4**; XXIX—Synthesis of Asymmetric Derivatives of 9-Carboxymethyl-3:6-diaminocarbazolemonosulphonic Acid. *Ibid.*, **714–6**; *Chem. Abs.*, **49**, 7546–7 (10 June 1955).

XXVI—Asymmetric N^{N'}-disubstituted derivatives (I) of 9-carboxymethyl-3:6-diamino-1:8-carbazole disulphonic acid (I) where the substituents (X and Y respectively) were C₆H₄NHCO (II), anilinohydroxytriazyl (III) and *p*-CH₃CONH C₆H₄CO (IV) have been made. I (X = II, Y = III) was obtained by treating 9-carboxymethyl-3-amino-6-phenylureido-1:8-carbazole disulphonic acid (V) first with cyanuril chloride and then with aniline. I (X = II, Y = IV) was obtained by acylation with *p*-nitrobenzoylchloride, reduction and finally acetylation with acetic anhydride. I (X = III, Y = IV) was similarly obtained from 9-carboxymethyl-3-amino-6-(4-hydroxy-6-anilino-*s*-triaz-2-ylamino)-1:8-carbazole disulphonic acid. They had a blue violet fluorescence of medium intensity under ultraviolet irradiation.

XXVII—Preparation of compounds of formula—



(X = C_6H_5NH , $o-CH_3C_6H_4NH$, $o-CH_3OC_6H_4NH$, $p-CH_3OC_6H_4NH$, $p-NaO_3SC_6H_4NH$, $o-NaO_3SC_6H_4NH$, $(CH_3)_2N$ or $HOCH_2CH_2NH$; Y = OH) is described. Under ultraviolet irradiation they show blue to greenish blue fluorescence of greater intensity than that shown by the corresponding derivatives from 9-carboxymethyl-3:6-diaminocarbazole disulphonic acid. They have good affinity for cotton.

XXVIII—Preparation of compounds of above formula where X = Y = C_6H_5NH , $o-CH_3C_6H_4NH$, $(C_6H_5)_2N$, $o-NaO_3SC_6H_4NH$ or H_2N ; or X = C_6H_5NH and Y = $o-NaO_3SC_6H_4NH$. Their blue to bluish-violet fluorescence has about the same intensity as those described in XXVII. Presence of a sulphonic group in the carbazole nucleus seems to affect the intensity and fluorescence wavelength. With these compounds and those described in previous papers the sodium salts show more intense fluorescence than the free acids and they show a better brightening effect on cotton.

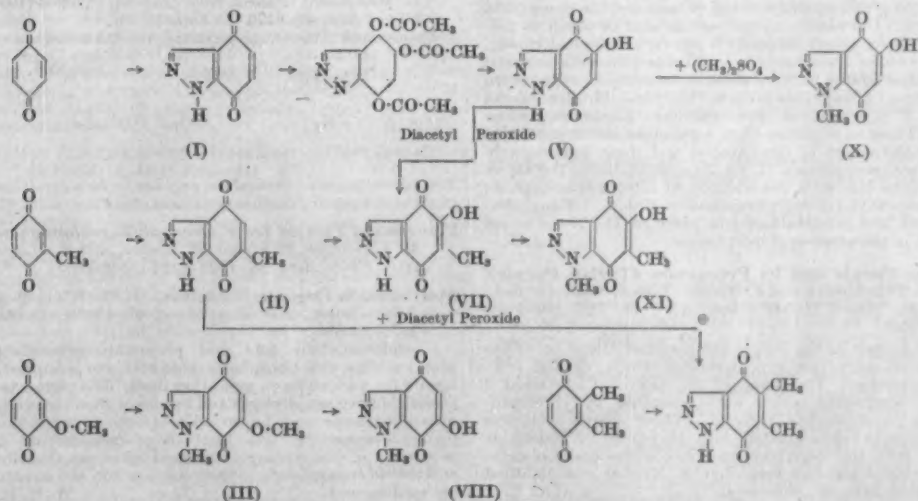
XXIX—Eleven derivatives of 9-carboxymethyl-3:6-diamine carbazolemonosulphonic acid (I) containing the following radicals asymmetrically have been made. When the mono-N salt of I is expressed as H_2NWNH_2 they were classified in three groups (A) $C_6H_5CONHWNH_2$ (X = a triazinyl radical having as substituents HO, $o-NaO_3SC_6H_4NH$; HO, C_6H_5NH ; C_6H_5O , C_6H_5NH ; or H_2N , H_2N) (B) $p-CH_3OC_6H_4NHCONHWNH_2$ (X = $CH_3ONHC_6H_4CO$ or a triazinyl substituted by HO, $p-NaO_3SC_6H_4NH$; HO, $o-NaO_3SC_6H_4NH$; or HO, C_6H_5NH) (C) $ZNHWNH[R(NHCH_2CH_2OH)NHC_6H_5]$ (R = trivalent triazinyl radical; Z = C_6H_5-NHCO , $o-CH_3OC_6H_4NHCO$ or $p-CH_3OC_6H_4NHCO$). Their blue to bluish violet fluorescence was stronger than that of the disulphonic derivatives and their brightening effect was also better not only for cotton but also for wool.

C. O. C.

Reaction of Diazomethane with Benzoquinones.

C. J. P. Spruit. *Rec. Trav. chim.*, **74**, 737-743 (June 1955).

By careful choice of reaction conditions, diazomethane and benzoquinone may be made to yield isindazole-quinones, together with an excess of dihydroxybenzodipyrzole (which was alone reported by previous workers); the reaction proceeds much more readily with subst. benzoquinones. The 4:7-dihydroxyisindazoles formed in the latter cases were unstable in air, and were thus converted immediately to the corresponding quinones. The following scheme has been carried out, and the proposed relationships are based on analogy with the known behaviour of the naphthaquinones and the thianaphthen-quinones—



Unsatisfactory analyses obtained in certain cases, and though this is discussed at length, no satisfactory explanation was reached. The Na salts of V and VIII were also prepared, and were of low solubility; they are derived from the OH rather than the NH acids.

J. W. D.

Perimidine Dyes and Intermediates. R. A. Jeffreys. *J.C.S.*, 2394-2397 (July 1955).

Intermediates and cyanine and merocyanine dyes, all possessing a perimidine ring, are described. The dihydropyrimidine ring system has strong electron-releasing powers indicated by the reversal of Kundt's rule in some merocyanines. An abnormal condensation between 2-methylperimidinium salts and ethoxymethylene-keto-heterocyclic intermediates gives a pair of dyes in each of four cases investigated, the lighter being a merocyanine, and the deeper a trinuclear merocyanine, which may be considered as a chain-substituted mesoionic oxonol.

H. H. H.

Disperse Dyes. IV—Synthesis of 1-Amino-4-hydroxyanthraquinone. (I). Y. Banaho and K. Kondo. *J. Chem. Soc. Japan, Ind. Chem. Sect.*, **57**, 751-3 (1954); *Chem. Abs.*, **49**, 9279 (10 July 1955).

The best method found is to stir urea (50), leuco quinizarin (24.2) and acetic acid (18) at 110-113°C. for 30 hr. in butanol (250) which yields 24.5 g. crude green crystals on vacuum distillation. It is purified by heating with 50-60% of sulphuric acid and diluting with water to give a 90% yield of the pure dye. Apparently the leuco quinizarin is aminated by the NH_2 produced by reaction between the urea and the butanol.

C. O. C.

Reaction of Indolylmagnesium Iodide with Enol-lactones of γ -Keto Acids. A. R. Katritzky and R. Robinson. *J.C.S.*, 2481-2485 (July 1955).

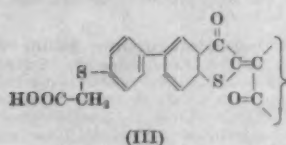
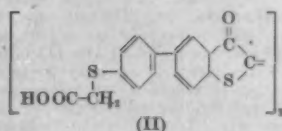
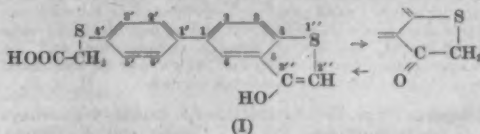
Indolylmagnesium iodide is found to react with 4:5-dihydro-5-oxo-2-phenylfuran, α -angelicalactone and γ -oxopimelic anhydride to give 1-acylated indoles. 1-Laeuloylindole is cyclised to 3-methyl-1:2(2':7')-benzylenepyrid-6-one, the action of alcoholic alkali on which supports an explanation previously advanced (cf. Bailey and Robinson, *Nature*, **161**, 433 (1948)) for the yellow colour and green fluorescence frequently encountered in alcoholic alkaline solution of certain strychnine deriv.

H. H. H.

Studies in Indigoid Dyes. XIV—Thioindigoid Dyes derived from Diphenyl-4:4'-disulphonic Acid. F. C. Dutta and D. Mandal. *J. Indian Chem. Soc.*, **32**, 339-343 (June 1955).

4:5-Diphenyl-(3'-hydroxyl-1''-thiophen)-4'-thioglycollic acid (I), prepared from diphenyl-4:4'-disulphonic acid via the disulphonyl chloride, dithiol, and dithioglycollic acid followed by cyclisation of one thioglycollic acid side-chain with phosphorus pentachloride and pyridine, on oxidation

with alkaline potassium ferricyanide yields the corresponding blue thioindigo (II). I condenses with the following diketones, in acetic acid containing some HCl, to give a series of vat dyes (III)—(a) Isatin (violet-red); (b) 5:7-dinitroisatin (chocolate); (c) *m*-bromoisatin (pinkish violet); (d) acenaphthenequinone (pinkish violet); (e) 5-nitroacenaphthenequinone (light blue); (f) 5-bromo-acenaphthenequinone (pink); (g) 5:6-dinitro-acenaphthenequinone (greenish grey); (h) phenanthraquinone (chocolate brown); (i) acenanthraquinone (copper red). A bluish-violet ethyleneindigo is obtained by condensing 2 mol. of I with glyoxal sodium bisulphite.



A. J.

Oxidation of Phenols with Copper-Amine Catalysts and its Relation to the Mode of Action of Tyrosinase. I—Catalytic Oxidation of Monohydric Phenols to *o*-Quinone Derivatives. W. Brackman and E. Havings. *Rec. Trav. chim.*, **74**, 937-955 (July 1955).

In a search for a model to replace tyrosinase in the oxidation of phenols to *o*-quinones it has been found that phenols and naphthols are readily oxidised by oxygen in the presence of the copper-ammonia or many copper-amine complexes. For more extended work the morpholine-cupric acetate complex was used. Red crystals of 4:5-dimorpholino-*o*-benzoquinone (I) were pptd. in 2-12 hr. according to conditions when O_2 was supplied rapidly to soln. of phenol, catechol, or guaiacol in methanol containing morpholine and cupric acetate—average yield = 60%. I is readily hydrolysed in acid or alkali to 2:5-dihydroxy-*p*-benzoquinone. When O_2 is supplied slowly, a 2% yield of 2:5-dimorpholino-*p*-benzoquinone is obtained and, after adding HCl, a stable copper complex of unknown structure. 2-Naphthol gives a 73% yield, but 1-naphthol only 37% yield, of 4-morpholino-1:2-naphthaquinone (II). There is evidence that *o*-quinones are first formed under the action of the catalyst and these subsequently react with morpholine. Thus, 2-naphthol, under the above conditions but with the addition of nitromethane, yields 4-nitromethyl-1:2-naphthaquinone (from 1:2-naphthaquinone and nitromethane) in place of II. No reaction occurs in the absence of morpholine.

A. J.

Murex Purple and its Precursors (Tyrian Purple). S. Bouchilloux and J. Roche. *Compt. rend. soc. biol.*, **148**, 1583-7 (1954); *Chem. Abs.*, **49**, 7757 (10 June 1955).

The extract of the purple dye gland of *Murex brandaris* contained only one prochromogen which yielded 6:6'-dibromoindigo. The gland of *M. trunculus* contained 2 promochromogens which were separated by chromatography in Amberlite IRC-50. These nearly colourless propigments when treated with purpurase or heated at 100°C. with HCl were converted into a blue and a mauve-red indigoid pigment respectively. Neither was identical with that from *M. brandaris*.

C. O. C.

Colour Reactions of Flavonoids with Beryllium Ions.

W. Bruns. *Naturwissenschaften*, **41**, 373-4 (1954); *Chem. Abs.*, **49**, 9225 (10 July 1955).

In presence of boric, oxalic or citric acid a number of flavonoids (flavonols: quercetin, myricetin, morin; flavone-3-glycosides: rutin, quercitrin, trifolin, myricitrin, robinin; flavonones: fustin, homocericodityol, "sakurammetin"; flavonone-7-glycosides: naringin, hesperidin; and isoflavones: tectoridin, iridin) form stable coloured complexes with Sb, Bi, Al, Pb, Cd, Cu, Zn, Fe and Be, their colour being intensified in an atmosphere of NH_3 . The Be complexes show strong fluorescence especially those with the flavonols and fustin, the fluorescence except for the isoflavones being green or yellow. The Be-fustin complex is stable to oxalic acid or 2*N*-HCl. The ultraviolet spectrum showed a distinct bathochromic shift of the long wave bands. In the 400-450 mμ range the Be complex showed stronger absorption than the flavonoid itself in methanol solution.

C. O. C.

Review of Research on Anthocyanins in Japan with Special Consideration of Natural Plant Pigments.

K. Hayashi. *Pharmazie*, **9**, 584-8 (1954); *Chem. Abs.*, **49**, 9745 (25 July 1955).

Review, 39 references.

C. O. C.

Antifungal Agent from Osage Orange Wood: Dyes present in the Wood.

R. A. Barnes and N. N. Geiber. *J. Amer. Chem. Soc.*, **77**, 3259-3262 (20 June 1955).

The wood of the Osage Orange, *Toxylon pomiferum*, contains approx. 1% of 2:3:4:5'-tetrahydroxystilbene which gives it its remarkable resistance to decay. Contrary to the statement by Mell (*Textile Colourist*, **53**, 749 (1931)) for which no experimental support can be found in the literature, Osage Orange wood contains no maclurin but only morin. Apparently Mell confused Osage Orange wood with Fustic for which its extract is sometimes used as a substitute.

C. O. C.

Colouring Matters of Actinomycetes. V—

Actinorhodin III. H. Brockmann and E. Hieronymus. *Chem. Ber.*, **88**, 1379-1390 (Sept. 1955).

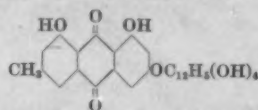
Mol. wt. determination on actinorhodin methyl ester suggests that it is a deriv. of dinaphthazarin (I). I itself is prepared in very poor yield by condensing 2:5:2':5'-tetramethoxydiphenyl with maleic anhydride and $AlCl_3$ -NaCl. I is obtained in dark red needles (benzene), decomp. above 270°C., converted by acetic anhydride into a yellow cryst. acetate, decomp. above 230°C. Absorption spectra of I and actinorhodin are very similar and so are those of their boracetates and their leuco acetates.

H. E. N.

Endothianin and Radicalisin—Two Pigments produced by *Endothia parasitica* and *E. radicalis*.

S. Shibata, O. Tanaka, G. Chihara, and H. Mitsuhashi. *Pharm. Bull. (Japan)*, **1**, 302-4 (1953); *Chem. Abs.*, **49**, 8229 (25 June 1955).

Endothianin, an orange pigment, has the structure—



The yellow pigment radicalisin appears to be a condensate of emodin with chrysophanol monoacetate.

C. O. C.

Fluorescent Pterins from *Drosophila melanogaster*.

M. Viscontini, E. Loeser, P. Karrer, and E. Hadorn. *Helv. Chim. Acta*, **38**, 1222-1224 (Aug. 1955).

Molybdenum Organic Pigments.

W. W. Williams and J. W. Conley. *Ind. Eng. Chem.*, **47**, 1507-1510 (Aug. 1955).

Phosphomolybdic acid and phosphotungstomolybdic acid, together with phosphotungstic acid, are precipitating agents for various basic and other dyes. The pigments so produced have less strength and brilliance than the original dyes, but possess much improved light fastness and valuable pigment properties. The methods of manufacture and properties of phosphomolybdic acid lakes are described, and the theories relating to their composition and structure are summarised.

W. K. R.

Molybdenum Inorganic Pigments. W. G. Huckle and E. Lalor. *Ind. Eng. Chem.*, **47**, 1501-1506 (Aug. 1955).

Molybdate Orange, the only important molybdenum-containing inorganic pigment, has a brilliant deep red-orange hue, high tinctorial strength, good opacity and permanency, and excellent application properties. It is a solid solution of lead chromate with 10-15 mol. % lead molybdate and 3-10 mol. % lead sulphate, and is prepared by co-precipitation. As the lead chromate is present in the unstable tetragonal form, the incorporation of suitable stabilisers is necessary in manufacture to prevent reversion to the stable monoclinic form with accompanying loss of valuable properties. The theory of the formation of the pigment, its properties, and its uses are discussed.

W. K. R.

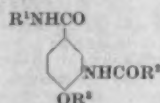
Recent Developments in Iron Oxide Pigments. I. C. Claro. *Offic. Digest. Federation Paint Varnish Production Clubs*, **26**, 902-916 (1954); *Chem. Abs.*, **49**, 7866 (10 June 1955).

Synthetic iron oxides are superior to the natural oxides in fineness, ease of dispersion, uniformity and hiding power. The particle size of the synthetic oxides is 1-3 μ . Introduction of carefully controlled grinding has made natural oxides available in particle size < 10 μ , whereas not long ago it was impossible to produce a natural oxide in which 25% of the particles were not > 15 μ . C. O. C.

PATENTS

Colour Couplers. Eastman Kodak Co. *USP* 2,694,635

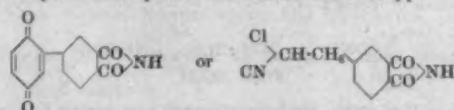
Compounds of formula—



(R¹ = coupler group free to react with an oxidised primary aromatic amino silver halide developer to form a dye; R² = a mononuclear acyl group of the benzene series containing an alkali solubilising group; R³ = satd. alk. of 10-25 C), e.g. 1-phenyl-3-(3'-chlorosulphonylbenzamide)-4'-dodecyloxybenzamide-5-pyrazolone, are colour couplers which yield dye images of improved absorption characteristics. C. O. C.

Substituted Phthalimides. FBy. *BP* 735,048

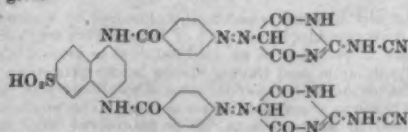
The diazo compound of 4-aminophthalimide or its N- β -hydroxyethyl derivative is condensed with a quinone or acrylonitrile to produce intermediates of the type—



useful in the preparation of phthalocyanines. Thus 4-aminophthalimide is diazotised by treating in aq. suspension with HCl and NaNO₂ at 8-15°C. The resulting solution is added dropwise into a suspension of benzoquinone in water containing acetic acid and sodium acetate at 20-23°C. The product, 4-phthalimido-benzoquinone, precipitates as a yellow powder. R. K. F.

Yellow Azo and Azoxy Direct Cotton Dyes containing Barbituric Acid Residues. FBy. *BP* 735,759*USP* 2,692,263

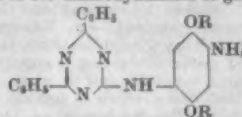
Yellow azo and azoxy direct cotton dyes are obtained by coupling diazo or tetrazo compounds of N-amino-benzoyl derivatives of suitable diazo components containing water-solubilising groups with barbituric acid or its derivatives formed by condensing malonic ester with urea, mono- or di-alkyl-ureas, guanidine, etc. Thus 1:4-bis(p-aminobenzoylamino)naphthalene-6-sulphonic acid tetrazotised and coupled with 2 mol. of cyanaminobarbituric acid gives—



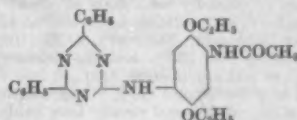
which dyes cotton dischargeable greenish yellow. E. S.

Blue Azoic Dyes. American Cyanamid Co.*USP* 2,688,616*USP* 2,690,437

Derivatives of 2:5-dialkoxyanilines of general formula—



(R = lower Alk) diazotised and coupled with arylides of 3-hydroxy-2-naphthoic acid give greenish blue azoic dyes and prints. Thus 2-chloro-4:6-diphenyltriazine and 4-amino-2:5-diethoxyacetanilide are refluxed in 50% aq. dioxan containing HCl to give—



which on hydrolysis gives the primary amino compound. A diazo soln. prepared from this amino gives greenish blue when used to develop cotton impregnated with 3-hydroxy-2-naphthanilide. E. S.

Direct Cotton Disazo Dyes for Aftercoppering. S. *BP* 733,747

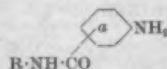
4 : 4'-Diamino - 3 : 3'-dihydroxydiphenylurea - 6 : 6'-disulphonic acid (I) is tetrazotised and coupled with *ortho*-coupling naphthols free of SO₃H and COOH groups to give direct cotton dyes for application by the aftercoppering process. Thus, 2-aminophenol-4-sulphonic acid is treated with phosgene to give benzene-1:2-oxazolone-4-sulphonic acid, which is nitrated and reduced to give the 5-amino compound, 2 mol. of which are condensed with 1 mol. of phosgene to give the urea. Treatment with aq. KOH then gives (I) by hydrolysis of the oxazolone groups. (I) is then tetrazotised and coupled with 2-hydroxy-6-naphthoanilide in presence of ammonia and pyridine, to give a red-violet aftercopperable dye for cellulose. E. S.

Dis- and Poly-azo Direct Cotton Dyes. S. *BP* 733,452

Two mol. of a mixture of two different aminoazo compounds free of groupings capable of metal-complex formation are condensed with 1 mol. of a dibasic halide X-CO-C(R):CH-CO-X (X = Cl or Br; R = H or CH₃) to give mixtures of symmetrical and unsymmetrical dyes whose dyeing and fastness properties on cellulosic fibres are in some respects superior to those of the similar symmetrical dyes of *BP* 701,709 (*J.S.D.C.*, **70**, 163 (1954)). Thus a mixture of 1.5 mol. of the aminomonoazo compound 2-naphthylamine-4:8-disulphonic acid \rightarrow m-aminacetanilide and 0.5 mol. of 4-aminoazobenzene-4'-sulphonic acid is dissolved in aq. NaOH and a soln. of 1 mol. fumaryl dichloride in benzene is added slowly together with aq. soda ash to maintain a weakly alkaline reaction. The product dyes cellulosic fibres greenish-yellow of good light fastness. E. S.

Azoic Dyes resistant to Plasticisers. FH. *BP* 733,160

Azoic dyes made by coupling a diazotised amine—



(R-NH-CO is *m*- or *p*- to the NH₂; R = H, Alk, aryl, or aralkyl; the benzene ring α may contain Alk, OAlk, or Hal as a substituent) with an arylide of 3-hydroxy-2-naphthoic acid (or of its 7-alkoxy- or 7-halogeno-derivatives), of 2-hydroxyanthracene-3-carboxylic acid, of 2-hydroxy-carbazole-3-carboxylic acid, of 3-hydroxy-diphenylene oxide (or sulphide)-2-carboxylic acid, or of 7:8-benzocarbazole-3'-hydroxy-2'-carboxylic acid, free of water-solubilising SO₃H or COOH groups, do not bleed into the plasticisers used with polyvinyl resins. Thus cotton cloth is first impregnated with a soln. in aq. NaOH of the 4-chloro-2:5-dimethoxyanilide of 3-hydroxy-2-naphthoic acid, and then developed in a bath containing a

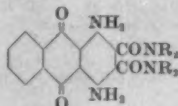
diazo soln. prepared from 3-amino-4-methylbenzanilide. The yellowish red azoic dyeing so obtained is coated with polyvinyl chloride plasticised with dioctyl phthalate, followed by heating for a few minutes at 170°C., to give a fabric of good fastness to rubbing and light. Or the previously prepared pigment, e.g. 3-amino-4-methoxybenzanilide → 3-hydroxy-2-naphthanilide, is incorporated into a thickened paste containing a copolymer of vinyl acetate and butyl acrylate and a water-soluble urea-formaldehyde condensate which is printed on cotton or viscose cloth, subjected to dry heat at ca. 110°C., and then coated with a waterproof layer as described above.

E. S.

NN'-Dihydroanthraquinone-azine from 1-Aminoanthraquinone. FBy. USP 2,693,469

A very good yield is obtained by melting 1-aminoanthraquinone (1 part by wt.) with an alkali phenolate (3-4) in presence of an oxidising agent and an alkali metal salt of a lower fatty acid. Thus 90% KOH (100) is added to phenol (150) at 100-120°C. and the product is mixed with anhydrous sodium acetate (40) at 160-180°C. To this is added over 30-60 min. 98% 1-aminoanthraquinone (100) and NaClO_2 (5-20) and finally iron oxide or MnO_2 (1). The melt is heated to 200°C. and stirred for 150-420 min. while gradually raising the temperature to 210°C. The whole is then baled in water (3000) for 1 hr. The resultant crude dye is filtered off, washed and dried. It is dissolved in conc. H_2SO_4 water added to bring the H_2SO_4 concentration to 83% whereupon the pure NN'-dihydroanthraquinone-azine is obtained in 70% yield (68.2 parts pure dye). If the filtrate is diluted a grey-brown precipitate results from which a further 9.6 parts of the dye can be secured by sublimation *in vacuo*. The total yield of dye is therefore about 77.5% calculated on the 1-aminoanthraquinone used. C. O. C.

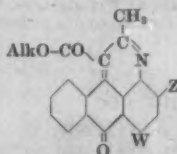
1:4-Diamino-2:3-anthraquinonedicarboxamides—Blue Dyes for Terylene. DuP. USP 2,692,272
Compounds of formula—



(R = same or different, H, alkyl or hydroxyalkyl, the total number of C atoms in both R's attached to the same N atom being < 5) dye polyethylene terephthalate fibres greenish blue of good fastness to light. Thus 1:4-diamino-2:3-anthraquinone dicarboxylic acid (17 parts), nitrobenzene (150) and thionyl chloride (42) were stirred and heated to 125°C., kept at 125°C. for 150 min., dried air passed through the soln. for 20 min. after which the soln. was cooled to 90°C. Ammonia gas was passed through at this temperature until no more was absorbed. The mixture was stirred at 90°C. for another hour, cooled to room temperature and alcohol (250) added to precipitate the dye. The dye was filtered off, washed with alcohol and hot water added. C. O. C.

Aza(3)-benzanthrone (Anthrapyridine) Dyes. S. BP 735,798

An aza(3)-benzanthrone of formula—

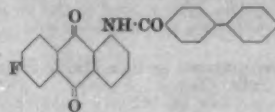


(W = Cl, Br, or NO_2 ; Z = H, Cl, Br, CH_3 or SO_3H) as described in BP 715,241 (J.S.D.C., 70, 584 (1954)) is condensed with a compound NHR^1R^2 (R^1 = H, alkyl or acyl; R^2 = H, alkyl, cycloalkyl, aralkyl, aryl, heterocyclic or azo compound radical), when the substituent W is replaced to give yellow to red dyes which may be further sulphonated. The unsulphonated products are useful for colouring lacquers, paper, or for "mass-dyeing" fibres; the sulphonated derivatives are dyes for cotton, wool, silk and superpolyamide and polyvinyl fibres. Thus 6-bromo-1-

carbethoxy-2-methyl-aza(3)-benzanthrone-4-sulphonic acid and aminocyclohexane are heated for 7 hr. at 90-95°C. in aq. Na_2CO_3 . R. K. F.

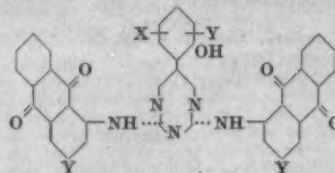
Fluorine-containing Acylaminanthraquinones—Vat Dyes. Ciba. BP 735,855

1-Amino-6-fluoroanthraquinone (BP 712,837, J.S.D.C., 70, 463 (1954)) is condensed with the acid halide of a diphenylcarboxylic acid, or alternatively 1-chloro-6-fluoroanthraquinone (BP 712,889, J.S.D.C., 70, 463 (1954)) with the amide of a diphenylcarboxylic acid; to give yellow vat dyes. Thus the dye—



is made by heating 1-amino-6-fluoroanthraquinone and diphenyl-4-carboxylic chloride (from the corresponding acid and SOCl_2) in nitrobenzene at 125-130°C. for 2 hr. R. K. F.

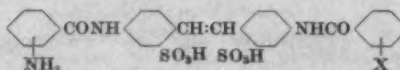
Halogenated Triazine Vat Dyes. American Cyanamid Co. USP 2,694,709
Dyes of formula—



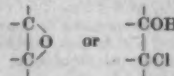
(X = Hal; Y = H or Hal), particularly those containing chlorine, are yellow vat dyes of good fastness to light which do not brown on fading and have very stable leuco compounds. They are obtained in very high yield as pure products if a 2-(o-hydroxyphenyl)-4:6-di-(a-anthraquinonylamino)-1:3:5-triazine is chlorinated or brominated so as to introduce halogen in the o-hydroxyphenyl group. C. O. C.

Fluorescent Brightening Agents. CFM. BP 734,433

Light yellow powders soluble in dilute alkali and showing bluish white fluorescence are obtained by treating compounds of formula—



(X = H or amino, the benzoyl groups may contain further substituents) with hydroxyalkylating agents containing the radical—



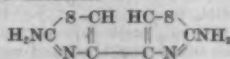
Thus 4:4'-di(4''-aminobenzoylamino)-stilbene-2:2'-disulphonic acid (60.8 parts) is stirred with ethylene oxide (88) in water (1000) at 100°C. in an autoclave until a sample taken cannot be diazotised. After cooling the precipitate is dissolved by adding Na_2CO_3 and the solution filtered. On acidifying and washing the precipitate with water a slightly yellowish powder is obtained which is readily soluble in dilute alkali and has affinity for cellulose, wool, polyamides, and polyurethanes. C. O. C.

7-Dialkylamino-4-methylcoumarin Salts—Fluorescent Brightening Agents. General Aniline. BP 735,395

The oily or tarry condensate obtained by condensing a m-dialkylaminophenol (Alk of 1-2 C) with ethyl acetate in presence of an acid catalyst is ground with a solid sulphonic acid having strong acidic properties, e.g. sulphamic acid or a naphthalene disulphonic acid, using 2-10 times as much acid as condensate. The product can be used in aqueous soln. as a fluorescent brightening agent for wool. C. O. C.

Phthalimide Derivatives of 2:2'-Diamino-4:4'-dithiazole—Direct Dyes. DuP. *USP* 2,692,267

Direct dyes are produced by condensing thio-phthalimide, iminophthalimide, *o*-cyanobenzamide, or their halogeno, methyl methoxy or nitro derivatives with 2:2'-diamino-4:4'-dithiazole—

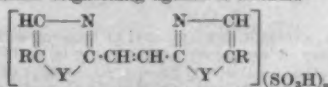


Thus 2:2'-diamino-4:4'-dithiazole (10 parts) and crude iminophthalimide (30) were stirred with ethylene glycol (220) and heated. After 3 hr. under reflux the mixture was cooled to 40°C., filtered and the press cake coated with ethyl alcohol. After oven drying at 90°C. a yellow dye (21) was obtained which was purified by dissolving in methyl alcohol-choline solution and reprecipitating with glacial acetic acid. It had the formula $\text{C}_{20}\text{H}_{12}\text{N}_8\text{S}_2\text{O}_2$. C. O. C.

Fluorescent Brightening Agent. CFM. *BP* 734,981

Modification of *BP* 722,543 (*J.S.D.C.*, 71, 197 (April 1955)).

Fluorescent brightening agents of formula—

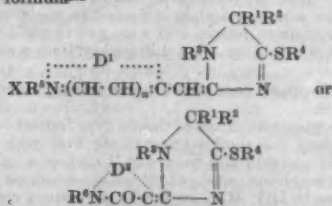


(R = residue of the benzene series; Y = O or S; n = 1, 2 or 3) are obtained by treating an aminoketone of formula $\text{RCOCH}_2\text{NH}_2$ with an acid or functional derivative thereof of formula $\text{X-CO-CH}_2\text{CH}_2\text{CO-X}$ (X = group exchangeable for amino, e.g. Cl), transforming the resulting acylamino ketone with a condensing agent and, if desired, with phosphorus sulphide, into the bisazole and then sulphonating. Thus fumaric acid chloride (80 parts) is dropped over 1 hr. into *o*-aminoacetophenone hydrochloride (172) and anhydrous sodium acetate (100) in water (1000) at 5–10°C. More anhydrous sodium acetate (100) is added and the mixture stirred for 1 hr. at 15°C. and then for 4–5 hr. at 20–25°C. The mass is then poured while stirring on to ice (2000), the colourless fumaroyl-di-(*o*-aminoacetophenone) which separates out filtered by suction and washed. It (10) dissolved in sulphuric acid monohydrate (80) is stirred for 1 hr. at 40–45°C. and then for 3 hr. at 70–75°C. The soln. is cooled, and poured while stirring on to ice (280). Excess H_2SO_4 is neutralised with CaCO_3 . It is filtered hot and the filtrate heated with Na_2CO_3 to yield a soln. of the Na salt of the sulphonic acid of 1:2-di-(5'-phenyloxazolyl-2'-)ethylene which has affinity for cellulose on which it shows intense blue fluorescence. C. O. C.

Cyanine Dyes containing an Iminoazolenine Ring.

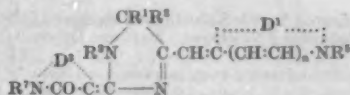
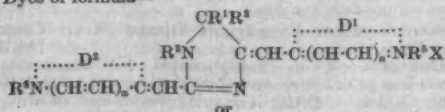
Ilford.

Dyes of formula—

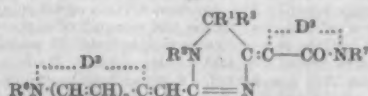


(R^1 and R^2 = hydrocarbon groups or together form an aliphatic carbocyclic system; R^3 and R^4 = identical Alk; R^5 = Alk; R^6 = hydrocarbon; X = acid radical; n = 0 or 1; D^1 = atoms to complete a 5- or 6-membered ring; D^2 = residue of a heterocyclic keto-methylene nucleus, or R^6 is linked via C and N to a C atom in D^2 to form a second heterocyclic nucleus), e.g. (4-methylthio-1:5:5-trimethyl-iminazolenine-2-)-(3-methylbenzothiazole-2')-monomethin cyanine perchlorate are photographic sensitizers. *BP* 734,793

Dyes of formula—

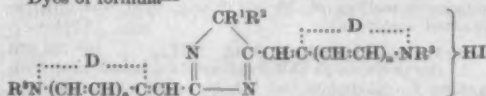


or



(R^1 and R^2 = Alk or aralkyl or together form an aliphatic carbocyclic system; R^3 = lower Alk; R^4 and R^5 = Alk; R^6 = hydrocarbon; X = acid radical; n = 0 or 1; D^1 and D^2 = atoms to complete a 5- or 6-membered ring; D^3 = atoms to complete a keto-methylene nucleus or R^7 is linked via C and N to a C atom in D^3 to form a second heterocyclic nucleus), e.g. [2-(1':2'-dihydro-1'-methylquinolinyldiene-2')-methyl-1:5:5-trimethyl-iminazolenine-4-](1''-methylquinoline-2'')-monomethin-cyanine iodide, are photographic sensitizers. *BP* 734,799

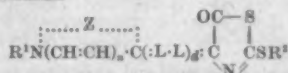
Dyes of formula—



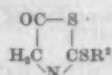
(R^1 and R^2 = Alk or together form an aliphatic carbocyclic ring; R^3 = Alk; n = 0 or 1; D = atoms to complete a 5- or 6-membered ring; in any one compound D, R^5 and n are the same in both cases), e.g. 2:4-di[(2':3'-dihydro-3'-ethylbenzothiazolydene-2')-methyl-5:5-dimethyl-iminazolenine hydroiodide], are photographic sensitizers. C. O. C.

merocyanine Dyes containing a 5(4)-Thiazolone Nucleus. Eastman Kodak Co. *USP* 2,692,829

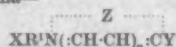
Dyes of formula—



(R^1 and R^2 = same or different Alk; n = 0 or 1; d = 0, 1 or 2; Z = atoms to complete a 5- or 6-membered nucleus; L = subst. or unsubst. methin), e.g. [2-(3-ethylbenzoxazole)] [4-(2-ethylthio-5(4)-thiazolone)] dimethinmerocyanine, are non-migrating, non-staining photographic sensitizers. They may be made e.g. by condensing a compound of formula—



with one of formula—



(X = anion; Y = an electronegative group). C. O. C.

Feed Preparation for Furnace Black Production. Phillips Petroleum Co. *USP* 2,693,441

Titanium Dioxide. National Lead Co. *BP* 734,448
A pigment which is particularly inert towards film-forming vehicles and resists chalking and discoloration is obtained by treating TiCl_3 , a silicon halide or oxyhalide vapour at 900–1050°C. with free oxygen and calcining the resultant oxide at 1100–1300°C. C. O. C.

Zinc Sulphide Phosphors. British Thomson-Houston Co. *BP* 734,652

A phosphor showing yellow and orange under ultraviolet radiation is obtained by heating ZnS with a copper salt (0.01–1.0% of Cu on the wt. of the ZnS) containing 0.3–1.0% of Cu on the wt. of the ZnS and 1–30 times as much Al as Cu. *BP* 734,653

An orange-red phosphor is obtained by heating ZnS with a copper salt (0.01–1.0% of Cu on the wt. of the ZnS) and heating in an atmosphere of H_2S for 30–60 min. C. O. C.

Concentrated Stable Solutions of Reduced Indophenol Sulphurised Vat Dyes. Southern Dyestuff Corpn.

BP 735,714

Stable, non-precipitating solutions of Hydron Blue or Indocarbon Black ready for immediate use in package dyeing are obtained by incorporating a small amount of a hydrotropic agent. The addition of this agent enables a dye liquor of twice or thrice the normal strength to be obtained. Thus sodium xylenesulphonate (25 parts) was dissolved in water (50) and to the solution Hydron Blue press cake (70) was added. Sodium hydrosulphide 40% (70) was added and the whole stirred while being heated to 90–100°C. After 1–2 hr. at this temperature the dye was completely dissolved. Undissolved matter was filtered off. The product is ready for use without addition of any further reducing agent in alkali. Thus cotton (100) was dyed dark blue in water (1500–2000) to which this solution (20) had been added. C. O. C.

Pigment Compositions for Colouring Rubber and Other Polymers. ICI. BP 735,534

Non-dusting compositions dispersing readily in rubber and other polymers are obtained by mixing a pigment with a polyvinyl methyl ether and a water-insoluble substance melting at 35–100°C., e.g. opal wax (hydrogenated castor oil). C. O. C.

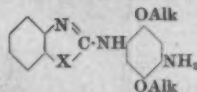
3-Hydroxy-2-naphthoic Acid. FH. BP 736,476

In the preparation of 3-hydroxy-2-naphthoic acid from sodium β -naphtholate and CO₂ under pressure, improved yields (42–47% instead of 32–37%) are obtained by carrying out the reaction in presence of an alkali hydride, e.g. ca. 12% sodium hydride calculated on the weight of β -naphthol. The alkali hydride may be produced *in situ* from the metal and hydrogen. E. S.

Azoic Diazo Components having Benzoxazole and Benzthiazole Residues. American Cyanamid Co.

USP 2,695,298

Amines—



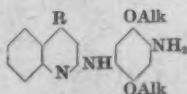
(X = O or S)

give greenish-blue pigments and azoic dyeings and prints when diazotised and coupled with suitable coupling components. Thus 4-amino-2:5-dimethoxyacetanilide is refluxed in 50% aqueous dioxan with 2-chlorobenzoxazole in presence of HCl, and the acetyl group removed by acid hydrolysis to yield the base (X = O; Alk = CH₃). When diazotised and applied to cloth padded with 3-hydroxy-2-naphthanilide it yields a blue. E. S.

Azoic Diazo Components having a Quinoline Residue. American Cyanamid Co.

USP 2,694,714

Amines—

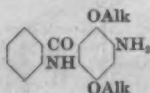


(R = H or CH₃) give greenish-blue pigments and azoic dyeings and prints when diazotised and coupled with suitable coupling components. Thus, 4-amino-2:5-diethoxyacetanilide is condensed with 2-chlorolepidine by refluxing in 50% aqueous dioxan in presence of HCl. Hydrolysis yields the amino compound (R = CH₃; Alk = C₂H₅), which when diazotised and applied to cotton padded with 3-hydroxy-2-naphthanilide gives a blue. E. S.

Aminoacridones—Azoic Diazo Components. American Cyanamid Co.

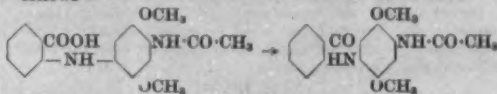
USP 2,694,713

Aminoacridones—



diazotised and coupled with arylides of 3-hydroxy-2-naphthoic acid give greenish blue pigments or azoic dyeings and prints, and a variety of other shades with appropriate coupling components. Thus 4-amino-2:5-dimethoxyacetanilide is acylated with *o*-bromobenzoic acid, and the product ring-closed by treatment in benzene first with

PCl₅ under reflux and then cold with SnCl₄ and HCl as follows—



Hydrolysis by refluxing with aq. alcoholic KOH gives the amine, which when diazotised and applied to cotton padded with 3-hydroxy-2-naphthanilide gives a blue. E. S.

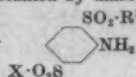
Violet and Blue Monoazo Disperse Dyes from 2-Amino-5-nitrothiazoles. Eastman Kodak Co.

BP 735,654

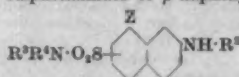
Similar to USP 2,683,708-0 (see J.S.D.C., 71, 556 (Sept. 1955)).

Red Monoazo Chrome Dyes. S. BP 735,128

Monoazo dyes obtained by diazotising amines—



(X = Alk, cycloalk, aralkyl, aryl of benzene series, NH₂, or a primary or secondary amino-group of < 15 C linked by its N atom to the SO₂ group; R¹ = aryl of benzene series containing OH and COOH *ortho* to each other) and coupling with sulphonamides of β -naphthylamine—



(R¹R²N³O₂S is in the 6- or 7-position; Z = H or OH when R¹R²N³O₂S is in the 6-position, and H when R¹R²N³O₂S is in the 7-position; R² = H, CH₃ or C₂H₅; R³ = H or Alk; R¹ = Alk, cycloalk, aralkyl, or aryl of benzene series) dye wool red from a weakly acid or neutral bath, the shade altering only slightly on afterchroming. Some are applicable by the metachrome process. Thus 2-amino-3'-carboxy-4'-hydroxy-4-methylsulphonyldiphenylsulphone is diazotised and coupled with 2-amino-8-naphthol-6-sulphon-N-ethylanilide in presence of methanol. The monoazo compound so formed dyes wool bright bluish red by the afterchrome or metachrome process. E. S.

Metal(Cobalt)-complex Monoazo Dyes for Wool, Nylon, etc. Gy. BP 736,034

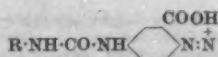
The cobalt complexes of *oo'*-dihydroxymonoazo compounds containing an SO₂Alk group but no SO₂H or COOH group dye wool, nylon, etc. from neutral or weakly acid baths. They are generally less deeply coloured and faster to light than the corresponding chromium complexes. Thus the monoazo compound 2-amino-4-methylsulphonylphenol \rightarrow acetacetanilide is heated to 80–90°C. with aq. cobalt acetate in presence of soda ash to yield the cobalt complex which dyes wool dull yellow from a weakly acid bath. E. S.

Metal(Copper)-complex Disazo Direct Cotton Blues. CFM. BP 735,879

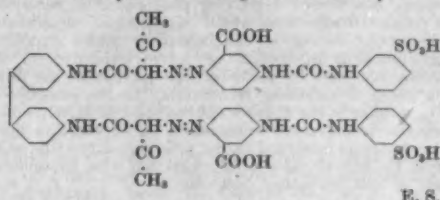
The copper complexes of disazo dyes formed by coupling tetrazotised 3:3'-dialkoxybenzidines first with 1 mol. of chromotropic acid and then with 1 mol. of a nitrogen-free naphtholsulphonic acid (which may contain as additional substituents OH, Hal, or OAlk) are direct cotton blues of better affinity and/or better light fastness than the copper complex of the symmetrical disazo compound dianisidine \rightleftharpoons (chromotropic acid), described in BP 407,684 (J.S.D.C., 50, 264 (1934)), and brighter in hue and of better affinity than the similar dyes of BP 613,939 (J.S.D.C., 65, 261 (1949)), in which the same components are used but the coupling components are applied in the reverse order. Thus, tetrazotised dianisidine is coupled first with chromotropic acid in presence of soda ash, and then with 1-naphthol-5-sulphonic acid in presence of NaOH. Heating at 85–95°C. with ammoniacal copper sulphate gives the blue copper-complex dye. E. S.

Greenish-yellow Copperable Disazo Direct Cotton Dyes. Gy. BP 736,166

One mol. of an unsulphonated *NN'*-diacetanetyl benzidine (which may be substituted in the 3:3'-position with Cl, Alk, or OAlk) is coupled with 1 mol. of a diazo compound—



(R = benzene or naphthalene series radical having at least one water-solubilising group) and 1 mol. of an α -carboxydiazo compound of the benzene or naphthalene series, which may be identical with the first diazo compound, to yield disazo dyes which give pure greenish yellows on cotton by the aftercoppering method. The diazo components are made by condensing R-NH₂ and 2-acetyl-amino-5-aminobenzoic acid with phosgene, or by first treating R-NH₂ with chloroformic phenyl ester and condensing the product R-NH-CO-OC₆H₅ with 2-acetyl-amino-5-aminobenzoic acid with elimination of phenol, followed by saponification of the acetyl-amino group. Thus, 2 mol. of 4-aminodiphenylurea-3-carboxylic acid-3'-sulphonic acid is diazotised and coupled with 1 mol. of NN'-diacetoacetylbenzidine to give the disazo dye—



E. S.

Metal(Copper)-complex Direct Cotton Dis- and Poly-Azo Dyes. BASF. BP 735,120

Diamines H₂N-R¹-X-R²-NH₂ (R¹ and R² = benzene series radicals containing no OH groups, and having at least one *ortho*-position to each NH₂ unsubstituted; X = a bridging atom or group) are tetrazotised and coupled with *ortho*-coupling phenols, naphthols or enolic compounds, and the products converted into copper complexes of *oo*-dihydroxyazo compounds by coppering under oxidising conditions using air in presence of a catalyst, or other oxidants such as H₂O₂, perborates, etc. The products, which must contain at least one SO₃H group, are direct cotton and leather dyes. Thus 4,4'-diaminostilbene-2,2'-disulphonic acid is tetrazotised and coupled with 2 mol. of R acid, and the disazo compound treated at 50°C. with CuSO₄ and H₂O₂ to give a pure blue for cotton and leather. E. S.

Red, Monoazo Pigments. Sherwin-Williams Co.

USP 2,694,055

Monoazo compounds dichloroanilinesulphonic acid → 3-hydroxy-2-naphthoic acid are converted into their Mn, Mg, Ca, Ba, Sr, Ce, Pb, Zn, or Cd salts which are red and maroon pigments. Diazo components in which the Cl atoms are *ortho* to each other give the best products. Thus 2,3-dichloroaniline-5-sulphonic acid is diazotised and coupled with sodium 3-hydroxy-2-naphthoate in presence of soda ash, and the product heated to 95°C. with aq. MnCl₂ to give a red pigment.

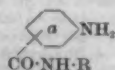
USP 2,694,056

Similar results are obtained using β -naphthol instead of 3-hydroxy-2-naphthoic acid. The Ca salt of the monoazo compound 2,3-dichloroaniline-5-sulphonic acid → β -naphthol for instance is similar in hue but superior in fastness properties to sodium lithol red (the sodium salt of the monoazo compound 2-naphthylamine-1-sulphonic acid → β -naphthol). E. S.

Monoazo Pigments. FH.

BP 734,285

Amines—



(CO-NH-R is *meta* or *para* to NH₂; R = H, Alk, Oalk, or Hal; the benzene nucleus α may contain substituents, e.g. Alk, Oalk, or Hal) are diazotised and coupled with esters of 3-hydroxy-2-naphthoic acid or its halogeno derivatives, to give pigments for dyeing polyvinyl chloride oranges, reds, and red-browns fast to bleeding. Thus the monoazo compound 3-amino-4-methoxybenzanilide → methyl 3-hydroxy-2-naphthoate is a red. E. S.

6-Acyl-2-naphthols. FBy.

BP 734,879

2-Hydroxy-1-naphthoic acid or 2-naphthol-1-sulphonic acid is heated with acid chlorides or anhydrides in presence of a Friedel-Crafts catalyst and the product heated with aq. mineral acid to split off the carboxyl or sulpho group, giving a 6-acyl-2-naphthol. Thus β -naphthol is treated in nitrobenzene at 0–5°C. with chlorosulphonic acid to give mainly the 1-sulphonic acid, which is added without isolation to a mixture of acetyl chloride and AlCl₃ in nitrobenzene, and heated to 70°C. for 8 hr. The mixture is poured on to ice and the crystals filtered off and hydrolysed in boiling aq. H₂SO₄. The product is freed from β -naphthol by salting out as its Na salt, redissolving and precipitating with HCl as free 6-acetyl-2-naphthol. E. S.

Fluorescent Brightening Agents for Use in Washing Nylon Goods (III p. 671).

Relationship between Crystal Structure and Habit-modifying Dyes (VIII p. 687).

Paper Chromatography of the Non-tannins of Tanning Extracts (XII p. 693).

Dye-sensitised Photopolymerisation of Vinyl Compounds (XIII p. 694).

Detection of an Unsubstituted *Para* Position in Phenols (XIV p. 695).

V—PAINTS; ENAMELS; INKS

Effect of Sunlight on Mixtures of Vegetable Oils and Pigments. F. A. Lee. *Nature*, 176, 463–464 (3 Sept. 1955).

Almond (I) and brazil nut (II) oils, obtained both by solvent extraction and by expression, were exposed to 10,000 ft.-candles of sunlight for 1 hr. The resulting peroxide value was increased tenfold in the case of I, and fivefold with II, in the presence of chlorophyll. Probably because of its reducing action, carotene retards the formation of peroxide, and the peroxide values of the carotene-treated oils were approx. equal to those of the pigment-free oils. Addition of mixtures of the two pigments led to a higher peroxide value for I, and either a lower or an equal value for II, than with the addition of carotene alone. J. W. D.

Adsorption of Surface-active Agents at Solid-Liquid Interfaces. J. L. Moilliet. *J. Oil & Col. Chem. Assoc.*, 38, 463–473 (Aug. 1955).

A review, mainly concerned with the wetting and dispersion of pigments in water and in oils. J. W. D.

PATENTS

Non-flaking Heat-resistant Coumarone Indene Aluminium Paint. Sheffield Bronze Paint Corp. USP 2,694,691

Addition to an aluminium paint based on a coumarone-indene resin of a calcium salt of an aromatic acid, e.g. calcium naphthenate, renders it non-flaking on iron even when heated to 1300–1700°F. C. O. C.

Vinyl Resin-Alkyd Resin Wrinkle Coating Composition. New Wrinkle. BP 734,664

A finely-divided vinyl resin is dispersed, preferably by grinding, in an alkyd type resin solution (which wrinkles with a drier). The resulting paste is mixed with colouring material, drier, solvent and more alkyd resin to yield a wrinkle coating composition which can be readily sprayed and baked. C. O. C.

Air-drying Wrinkle Finish Coating Composition. Council of Scientific & Industrial Research.

BP 734,576

Compositions which yield a regular wrinkled film on air drying are obtained by treating a vegetable oil containing glycerides of conjugated double bond acids, e.g. Kamala seed oil, with a natural or synthetic resin or an ester gum at 175–280°C. till a thread of 6–10 in. can be drawn from a drop of the cooled cooked material taken between the fingers. C. O. C.

Bactericide for Emulsion Paints. Dow Chemical Co. BP 732,490

Emulsion paints are protected against deterioration by micro-organisms by the addition of 0.01–0.05% of a haloacetamide, e.g. chloroacetamide. E. C.

Anti-misting Printing Inks. J. M. Huber Corpn.

BP 732,414

Reduced misting of conventional printing inks used in high-speed printing is obtained by dispersing in the water-immiscible ink approx. 5% of a bentonite and approx. 15% water.

E. C.

Metal-free Organic Driers (III p. 672).

Flow and the Critical Pigment Volume in the Paint Industry—Measurement of Oil Absorption by Pigments and Critical Pigment Volume (XIV p. 696).

VI—FIBRES; YARNS; FABRICS**Production and Utilisation of Non-woven Fabrics.**

H. L. Leventhal. *Amer. Dyestuff Rep.*, 44, P 464-P 466 (4 July 1955).

Electrostatic Charges on Fabrics. H. T. McLean.

Amer. Dyestuff Rep., 44, P 485-P 489 (18 July 1955).

Mechanisms of the development of static are briefly discussed, and the principles of the measurement of static described with particular reference to a fabric potential meter.

J. W. B.

Static Electrification of Filaments—Experimental Techniques and Results. S. P. Hersh and D. J. Montgomery.

Text. Research J., 25, 279-295 (April 1955).

The generation of charge on fibrous materials was studied using an apparatus by which one fibre was held fixed while a second was rubbed across it. Reproducibility was usually within 5% when the same two fibres were rubbed repeatedly. The charge generated depended upon the manner of rubbing, and the magnitude of the charge was directly proportional to the length of the material rubbed and to the normal force between the fibres (although in some cases a maximum limiting value was reached), but was independent of the apparent area of contact between the fibre and of the tension on the fibres. The effects produced by changes in velocity are more complex. Charge transfer was found to be independent of velocity when insulators (except Teflon) were rubbed together. For metals on insulators (except Teflon) the charge generated increased linearly with velocity until a limiting value was reached, after which it remained constant. When Teflon and metals were rubbed together, the charge increased linearly with velocity without reaching a maximum. When Teflon was rubbed with insulators, the charge increased linearly with velocity in some cases but remained constant in others. A triboelectric series was established from a study of the dependence of the sign and amount of charge transferred on the nature of the materials rubbed together. For metals on insulators the amount of charge generated was related to the work function of the metal and the position of the insulator in the triboelectric series. For insulators rubbed on insulators the amount of charge transferred appeared to be independent of the positions of the insulators in the series.

W. P. M.

Effect of Voltage and Time on the Electrical Resistance of Cotton. (a) G. E. Cusick and J. W. S. Hearle;

(b) S. P. Hersh and D. J. Montgomery. *Text. Research J.*, 25, 563-567 (June 1955).

(a) Observations are made upon the findings of Hersh and Montgomery (*Text. Research J.*, 22, 805 (1952)) that cotton yarn is ohmic in the range 50-2000 v., in apparent contradiction to the results of other workers. Various factors are suggested as contributing to the apparent contradictions, and the authors conclude that the ohmicity was mainly due to the length of time for which the voltage was applied; but it is doubtful whether the results are fundamentally significant, as they may not refer to an equilibrium state.

(b) The authors reply that there is no inconsistency in their results, as they, too, observed the time-dependent effects observed by Cusick and Hearle for resistances of the order of 10^{10} ohms (as used by Cusick and Hearle); but most of the work was carried out with higher resistances, some being of the order of 25×10^{10} ohms. However, there may be some inaccuracy, due to the use of a kind of hot-wire anemometer. Unless heat is lost quite rapidly from the filament, the measurements at different voltages or times are not isothermal when using this instrument.

This effect is usually negligible with high-resistance material, but it may become appreciable with better conductors (resistances of 10^{10} ohms or less). The effect of this phenomenon on the results is discussed.

W. P. M.

Degradation of Viscose Rayon Textiles by Light.

T. N. Kleinert and V. Mössner. *Textil-Rund.*, 10, 353-359 (July 1955).

The light degradation of bright and delustrated viscose rayon materials and of cotton has been studied by exposure to sunlight and, to a lesser extent, to the light from a quartz lamp. The degree of degradation was measured by the fall in tensile strength and the reduction in D.P. Degradation increases as the diameter of the fibres decreases, and delustrated fibres are more degraded than bright fibres of the same denier. In the case of viscose rayon fibres delustrated with TiO_2 , the nature of the degradation is complex. In certain circumstances the pigment may promote a series of degrading photochemical reactions, the combined effects of which can produce considerable degradation during a comparatively short exposure. Degradation is intensified by acidic impurities in the material and also by acidic groups present in the fibres as a result of oxidising treatments. In the presence of TiO_2 , sulphur in the fibres is oxidised to free H_2SO_4 , which gives rise to extensive degradation on exposure to light. Degradation is increased in the presence of metallic salts, e.g. of Cu and Cr, and after treatment with oxygenated washing and bleaching materials.

J. C. F.

Photochemical Degradation of Delustrated and Undelustrated Cellulosic Fibres—II. A. Agster.

Melliand Textilber., 36, 1-4 (Jan. 1955).

Oxidising agents and fluorescent brightening agents do not influence the degradation, which occurs at the 1:4-glucoside link. Aldehyde groups in matt fibres are catalytically oxidised to carboxyl groups by TiO_2 . The degrading agents may be H_2O_2 and atomic oxygen.

S. R. C.

Related Chemical Components of Some Bast Fibres.

M. Lüttke. *Melliand Textilber.*, 36, 763-765 (Aug. 1955).

(1) Ramie, (2) flax, (3) hemp, (4) jute, (5) *Hibiscus cannabinus*, and (6) sisal were examined for cellulose pectin, pentosan, wax, and lignin contents. The results are tabulated, and the fibres grouped in four classes—(1), (2)+(3), (4)+(5), and (6).

S. R. C.

Crease Recovery of Fabrics. I—Some Tensile Properties of Wool, Vicara, and Rayon Fibres and their Relationship to Fabric Crease Recovery.

J. F. Krasny and A. M. Sookne. *Text. Research J.*, 25, 493-498 (June 1955).

A study has been made of some single-fibre properties of wool, rayon, and Vicara zein fibres at various relative humidities, and the crease recoveries of fabrics made from the same fibres have been measured under identical conditions. In general, the wool and Vicara are similar in many load-extension properties. A fair correlation was found between fabric crease recovery on the one hand, and fibre tensile and work recovery on the other hand, at low and normal humidities. At high humidity, and particularly with wet fabrics, however, this relationship no longer holds. A general correlation also seems to exist between fabric crease recovery and work returned by stretched fibres during retraction from extension to 4%.

W. P. M.

Crease Recovery of Fabrics. II—Effect of Construction on Crease Recovery of Fortisan Fabrics.

J. F. Krasny, G. D. Mallory, J. K. Phillips, and A. M. Sookne. *Text. Research J.*, 25, 499-506 (June 1955).

An attempt has been made to establish some general relationships between crease recovery and variables of fabric construction by an investigation of 174 systematically varied Fortisan filament fabrics. Crease recovery is generally improved by open construction, i.e., for any one fabric weight, by employment of high yarn denier and wide yarn spacing, and of open weave, i.e. one with long floats. In the case of asymmetrical weaves (e.g. 2/1 or 5/1 twills), the crease recovery is higher when measured with the longer float outside the crease. In general, little effect on crease recovery was found if the yarn properties were varied only in the weft direction.

W. P. M.

Side-chain Orientation in Fibrous Proteins. R. D. B. Fraser. *Nature*, 176, 359-359 (20 Aug. 1955).

The 5150 cm^{-1} infrared absorption band can be completely eliminated from the spectra of fibrous proteins by drying at 105°C. for 3 hr., enabling the 5050 cm^{-1} band to be studied in some detail. The assignment of the 5050 cm^{-1} band to side-chain amide groups is confirmed by its relative prominence in silk, wool, and gliadin. The strong perpendicular dichroism of the side-chain amide band in wool indicates a tendency for the $-\text{CO}-\text{NH}_2$ groups to be oriented with the line joining the two H atoms perpendicular to the fibre axis. The dichroism of the side-chain amide band vanishes in hair extended 90% in steam, possibly because of random orientation of the H-H direction in the extended condition. Reaction of dry wool with D_2O reduced the optical density of the peptide band to half its original value, while the amide band was no longer detectable. Assuming that the fraction inaccessible to D_2O is crystalline, the side-chain amide groups must be concentrated in the amorphous regions but at points where there is considerable preferred orientation of chains parallel to the fibre axis. In view of the complete exchange of amide groups the assumption that there is a uniform distribution of side-chains throughout crystalline and amorphous regions does not seem to be justified.

W. R. M.

Studies on the Structure of Keratin. I—The Analysis of Fractions isolated from Wool oxidised with Peracetic Acid. C. Earland and C. S. Knight. *Biochim. Biophys. Acta*, 17, 457-461 (Aug. 1955).

A number of preparations of ammonia-soluble and -insoluble fractions obtained from wool oxidised with peracetic acid have been analysed for C, H, N, and S. Statistical examination of the results has shown that these fractions differ in C, H, and S contents, but that their N contents are identical. It is also shown by direct analysis that wool oxidised with peracetic acid does not form an ammonium salt under conditions where such a salt is formed with cysteic acid. It seems probable that strong links such as $-\text{SO}_3^- \cdots ^+\text{NH}_2-$ prevent the sulphonic acid residues in oxidised wool from taking part in normal reactions.

P. G. M.

Infrared Spectrum of Peracetic-acid-treated Wool. G. J. Weston. *Biochim. Biophys. Acta*, 17, 462-464 (Aug. 1955).

Infrared spectra in the range 1400 cm^{-1} -900 cm^{-1} are presented for virgin wool, peracetic-acid-treated wool, and L-cysteic acid. Spectral evidence, using established correlations between absorption frequencies and atomic groupings, has been obtained in favour of the direct formation of sulphonic acid groups and not an intermediate sulphonamide as a result of treating wool with peracetic acid.

P. G. M.

Reactivity of the Cystine Linkages in Wool towards Reducing Agents. A. J. Farnworth. *Biochem. J.*, 60, 626-635. (Aug. 1955).

The reactivity of the cystine linkages in wool towards reducing agents has been investigated before and after modification of the wool in various ways.

Reduction and alkylation of part of the cystine (the (A+B) fraction) allows the remainder (the (C+D) fraction) to react with sodium bisulphite and thioglycolic acid under conditions where it is normally inert. The reaction products are then the same as normally obtained with fraction (A+B). In contrast with this, reduction followed by cross linking of the thiol groups from fraction (A+B) through a methylene bridge does not allow fraction (C+D) to show this induced reactivity. Acetylation of polar side chains in wool leads to a general reduction in the reactivity of cystine. Treatment of wool with phenol isocyanate leads to blocking of all the polar side chains without affecting the cystine. Such treatment almost completely inhibits subsequent reactivity of the cystine linkages. It is suggested that the degree of reactivity of cystine linkages is largely governed by their accessibility to a reagent, close proximity of bulky side chains reducing reactivity through steric hindrance. Other factors such as varying structural arrangements may be responsible for the variation in the nature of the products resulting from a particular reaction.

P. G. M.

Amino Acid Composition of Normal Wools, Wool Fractions, Mohair, Feather, and Feather Fractions. W. H. Ward, C. H. Binkley, and N. S. Snell. *Text. Research J.*, 25, 314-325 (April 1955).

The variation in composition of normal wools of different breeds and geographical origins, mohair, and feathers was assessed by means of amino acid analysis. The mohair sample was found to be similar to wool in composition, the most striking apparent chemical differences being the relatively low sulphur and tyrosine contents and the relatively high value for aspartic acid of the mohair. White leghorn chicken feather keratin yielded larger amounts of cystine and glutamic acid than did wool. Heterogeneity of wool and feather keratins was shown by differences in amino acid composition of fractions prepared by treatment with acid, by mechanical separation, or by partial dissolution in aqueous alcohol. Feather differs from wool in having readily separable fractions differing greatly in content of histidine, lysine, and methionine. These fractions also differ appreciably in their content of several other amino acids. The authors compare their results with those of several other workers.

W. P. M.

Amino Acid Compositions of Several Wool Fractions as determined by Paper Chromatography. R. L. Golden, J. C. Whitwell, and E. H. Mercer. *Text. Research J.*, 25, 334-342 (April 1955).

A paper-chromatographic study is described of the amino acid composition of the epicuticle, cortical cell membranes, paracortex, and regenerated oxidised keratin of wool. The amino acid contents of the hydrolysates resulting from hydrolysis with hydrochloric acid were identified and estimated quantitatively by a modified two-dimensional paper-chromatography technique. The epicuticle was found to contain normal amounts of cystine but extraordinarily large quantities of aspartic acid and glutamic acid. Large amounts of the basic and small amounts of the dicarboxylic amino acids were found in the orthocortex. The paracortex was shown to contain considerably more cystine than the orthocortex. The extremely low total amino acid content found in the cortical cell membranes indicated the possible presence of some non-protein constituent.

W. P. M.

Effect of Non-uniformity of the Cross-sectional Area upon the Tensile Behaviour of Wool Fibres. E. C. Banky and S. B. Slen. *Text. Research J.*, 25, 359-361 (April 1955).

The extremely wide range in relative extension and the considerable scatter in Hookean slope, which are apparent when wool fibres are stretched, are explained on the basis of the non-uniformity of the cross-sectional area (C.S.A.) of the fibres under test. The C.S.A. of adjacent segments of wool fibres were measured and found to vary considerably. Experiments in which the C.S.A. of the fibre was measured at various points, and the elongations of various segments were measured when the fibre was extended, showed that differential elongation occurred, and this was particularly pronounced in the regions of low extension. At the same moment different portions of the fibre were in different stages of their elongation, causing an overlapping of the regions of low and high extension. The elongation of the thinnest portions of the fibre was greatest. Thus the irregular C.S.A. of wool fibres has a considerable effect on the load-elongation curve and its parameters. The load-elongation curve as registered by the dynamometer is the superimposed product of a varying number of individual curves, and the true stress-strain properties of the wool are represented by these individual curves.

The component curves will be superimposed with a subsequent delay due to the greater initial resistance of the wool to extension. The delay depends upon the magnitude of the differences in the C.S.A., and changes in the characteristics of the curve result. An apparent decrease in both extension to break and breaking stress occurs, and if no degradation is present, the thinnest segment of the fibre acts as a limiting factor for both characteristics.

W. P. M.

Influence of Electrode Potentials on the Extent of Reduction of Cystine in Hair Fibres by Reducing Agents. H. E. Jass and L. S. Foadick. *Text. Research J.*, 25, 343-353 (April 1955).

The effect of reducing agents on human hair fibres was measured by studying the relaxation curves of fibres kept

at constant elongation in a solution of the reagent. Where reduction occurred, the extent of reduction, as measured by the final equilibrium stress level, depended on the electrode potential of the reagent. The decrease in stress corresponded to reduction of cystine disulphide linkages within the fibre as shown by chemical analyses of treated hair fibres. The dependence of the amount of reduction on the reagent potential indicates that the cystine residues within the keratin fibre exhibit dissimilar E° values.

W. P. M.

Effect of Nuclear Radiation on the Wool Fibre. R. D. Kirby and H. A. Rutherford. *Text. Research J.*, **25**, 569-570 (June 1955).

Attention is drawn to a forthcoming publication in which the effect of nuclear radiation (principally neutrons and γ -rays) on the properties of wool will be described. Results of experiments wherein fibres were exposed to radiation are mentioned. Work completed to date shows that work exposed to radiation of up to 10^{16} nvt. was not appreciably affected; but at this level the alkali solubility started to increase, and the stress-strain properties of single fibres changed significantly. As the dosage increased beyond this level, there was a considerable change in many properties, and at 10^{16} nvt. the fibrous characteristics of the wool were completely destroyed. The effect on the 30% index is discussed, and from the results it is suggested that the predominating effect of nuclear radiation on wool, when exposed in an atmosphere containing oxygen and water, is one of oxidation of cystine; there may be some cleavage of amide linkages.

W. P. M.

Calorimetric Determination of the Heat of Reaction of Hydrochloric Acid and Wool. A. N. Derbyshire and R. H. Peters. *Trans. Faraday Soc.*, **51**, 1081-1090 (Aug. 1955).

Measurements have been made in the absence of added salt and in the presence of 0.19 M NaCl. The first absorption of acid occurs with negligible heat change, after which the heat produced varies linearly with the acid combined. Less heat is evolved in the presence of salt. Results in absence of salt are interpreted in terms of free and salt-linked carboxyl groups. It is postulated that the first absorption of acid occurs by combination of hydrogen ions with free carboxyl groups followed by absorption of acid by salt links, each link being broken and its constituent acid and basic groups combining with a chloride and hydrogen ion respectively with evolution of heat. The heat of reaction is -3.0 kcal./mole. The results in the presence of salt are consistent with the hypothesis that immersion of wool in salt solution causes breakdown of some salt links.

W. R. M.

Iodination of Wool. H. R. Richards and J. B. Speakman. *J.S.D.C.*, **71**, 537-544 (Sept. 1955).

When wool is iodinated with aqueous solutions of iodine in potassium iodide at different pH values and temperatures, reaction with the tyrosine side-chains is incomplete, even under the most favourable conditions. With alcoholic solutions of iodine, however, 96% of the tyrosine is converted into 3,5-di-iodotyrosine in 72 hr. at 22°C., and there is no combination with either histidine or tryptophan.

The fact that tyrosine can be iodinated exclusively and almost completely by such a simple procedure affords a useful means of discovering whether the lysine and tyrosine side-chains are cross-linked when wool is treated with formaldehyde. Since the amount of iodine which combines with the formaldehyde-treated wool, after loosely held formaldehyde has been removed with phosphoric acid, is the same as for untreated wool, it is concluded that the lysine and tyrosine side-chains are not cross-linked by formaldehyde. It has been shown also that the distribution of tyrosine along the length of single fibres can be determined by taking radiophotographs after the fibres have been iodinated with 131 I.

AUTHORS

Interaction of Acid-treated Wool and Silk with Water Vapour and Water. A. F. Caccia and H. J. White. *Text. Research J.*, **25**, 506-516 (June 1955).

Extensive results are given on the effects of hydrochloric acid and picric acid on the capacity of wool to absorb water. Both the effect of changing the relative humidity and the effect of changing the acid content of the fibres were studied. Less extensive results were obtained using sulphuric acid, oxalic acid, and Orange II

acid and salt on wool, and hydrochloric acid on silk. Collateral measurements were also made on the swelling of human hair in picric acid solution and on the electrical resistance of wool fibres containing acid at various relative humidities. The absorption of acid decreases the capacity of wool to absorb water at low and moderate relative humidities. The efficiency with which the absorption of water is reduced depends on the identity of the acid. At the highest relative humidities the acids divide into two groups. In the case of the first group, of which picric acid is an example, the effect of acid on the absorption of water is the same as at lower humidities. In the case of the second group, of which hydrochloric acid is an example, there is a second mechanism of interaction, which tends to increase the absorption of water and competes with the mechanism causing the decrease at lower relative humidities. The second mechanism may become dominant, causing a net increase. This has been shown by direct measurement and by analysis of swelling measurement.

The mechanism of the interaction decreasing the absorption is identified as competitive absorption of acid on primary bonding sites for water. The mechanism of the interaction increasing the absorption has been tentatively identified as osmotic absorption of water resulting from mobility of absorbed anions at the highest relative humidities. It is also possible that anion hydration is important in some cases.

W. P. M.

Adsorption of Some Organic Vapours by Wool and Nylon. H. R. Chipalkatti, V. B. Chipalkatti, and C. H. Giles. *J.S.D.C.*, **71**, 652-660 (Nov. 1955).

Vapour-phase adsorption experiments have been made with a number of organic compounds on nylon and wool fibres. Preliminary tests showed that all hydroxylic compounds have very considerably greater affinity for the fibres than have other polar substances. Rate curves and/or isotherms have been determined for acetic acid, ethanol, methanol, phenol, and other compounds. It is shown that in absence of air and moisture the fibres are stable for long periods at temperatures as high as 150°C., so that adsorption tests can be made with substances of high boiling point, e.g. β -naphthol. At these temperatures each successive adsorption-desorption cycle produces a rise in the adsorptive capacity of the fibre, and sometimes in the rate of diffusion of the adsorbate. The evidence is consistent with a mechanism by which hydroxylic compounds are adsorbed as a monolayer on localised sites, probably by hydrogen bonding at the oxygen atoms of the amide or peptide groups.

AUTHORS

Estimation of Alkali Solubility of Silk Fibroin.

M. Oku and I. Shimizu. *Textil Praxis*, **10**, 551 (June 1955).

Details are given of a method of estimating the alkali solubility of silk fibroin. The relationship between the alkali solubility and fibre quality and also variations in this property in different portions of the silk fibroin are indicated. Treatment of silk fibroin with formaldehyde results in appreciable decrease in alkali solubility. The alkali solubility of undamaged wool is somewhat higher than that of silk.

B. K.

Improving the Quality of Silk by Chemical Treatment. II—Treatment with Paraformaldehyde. M. Nakashini. *J. Sericult. Sci. Japan*, **23**, 261-4 (1954); *Chem. Abs.*, **49**, 9933 (25 July 1955).

Treatment with paraformaldehyde increases the resistance of silk to alkali and light and decreases its strength, extensibility and affinity for dyes.

C. O. C.

Amino Acid Composition of Bombyx mori Silk Fibroin and of Tussock Silk Fibroin. W. A. Schroeder, L. M. Kay, B. Lewis, and N. Munger. *J. Amer. Chem. Soc.*, **77**, 3908-3913 (20 July 1955).

Chromatographic analysis of the amino acids present in *Bombyx mori* silk fibroin and in Tussock (*Antheraea pernyi*) silk fibroin showed marked differences in composition. Glycine, alanine, serine and tyrosine make up 90% of the residues in both fibres but there is almost exact reversal of the amounts of glycine and alanine in one fibroin compared with the other. The remaining 10% in Tussock silk contains more histidine, arginine and aspartic acid and less threonine and valine than *Bombyx mori* silk.

C. O. C.

Pilling. Anon. *Bull. Inst. Text. France*, (54), 65-71 (June 1955).

A review of the following aspects of pilling—manifestation, mechanism, methods of measuring pilling tendency, and methods of reducing the tendency of fabrics to pill. J. C. F.

Physicochemical Behaviour of Synthetic Polyamide Fibres. W. Schefer. *Textil-Rund.*, 10, 279-296 (June), 365-374 (July), 423-427 (Aug. 1955).

A study of 6,10-nylon (hexamethylenediamine-sebacic acid) (I), 6,6-nylon (hexamethylenediamine-adipic acid) (II), and 6-nylon (ϵ -caprolactam) (III). Components liberated by HCl hydrolysis were separated on paper chromatograms in *n*-propanol-25% ammonia-water (60:30:10) and detected by spraying with 0.04% aqueous Bromothymol Blue containing NaOH (I and II) or ninhydrin (III). *R_F* values are listed. Hot-water Soxhlet extraction for 8 hr. gave < 1% weight loss for all three fibres. Data on average dimensions and tensile properties are given for original and extracted samples. Regains (20°C., 65% R.H.) determined after previous intensive drying of extracted samples were—I 2.04%, II 4.24%, III 4.54%, in agreement with the smaller number of polar sites in I. Ash contents of all three extracted samples were < 0.1%. Load-extension diagrams are given for original and extracted samples. After extraction, I showed unaltered elongation at break (a) and slightly smaller breaking strength (b). For II, both (a) and (b) were somewhat reduced. For III, (a) was unaltered, but (b) considerably greater. Molecular weights and degrees of polymerisation were measured viscosimetrically for solutions of I, II, and III in *m*-cresol. Amino end-groups of I, II, and III were determined by conductimetric titration of a *m*-cresol-ethanol solution with ethanolic HCl in a stream of pure N₂; carboxyl end-groups of III by conductimetric titration with ethanolic NaOH; and carboxyl end-groups of I and II by titration of a solution in benzyl alcohol with NaOH in benzyl alcohol-ethanol using phenolphthalein as indicator. For I the mol. wt. from the end-group determination agreed well with the viscosimetric value. For II and III the values from end-group determination were higher than the viscosimetric values. In the case of II this is probably due to thermal modification, since it must be spun at a higher temperature than I. The discrepancy in the values for III is probably due to the blocking of NH₂ end-groups by stabiliser molecules. Previous work on end-group determination on polyamides is fully reviewed. The effects of progressive hydrolysis of I, II, and III with 3*N*-H₂SO₄ at 50°C. on end-groups, viscosimetric behaviour, and load-extension relations are described, and the structural significance of the results is discussed.

The relaxation at a constant extension of 10% has been studied. With the fibres immersed in anhydrous benzene the load for constant extension (*K_t*) falls sharply and then more slowly, reaching a constant value after 15 hr. The curves for the three fibres have a similar shape, the values of *K_t* being greatest for I and least for III. When fibres which have been relaxed in benzene are transferred to water, *K_t* again decreases, reaching a limiting value *K_∞* within 500 min. If $P_t = K_t - K_\infty$, then a plot of $\log P_0/P_t$ against *t* is linear, and the slope *k* may be taken as representing the velocity constant of the reaction between water molecules and the hydrogen bonds of the fibres. From measurements at 20°C. and 30°C., *k* was found to be largest for III, somewhat smaller for II, and remarkably small for I. The activation energies of the reaction were found to be 9400, 7100, and 9000 cal./mole for I, II, and III respectively. For fibres relaxed in benzene and subsequently transferred to absolute alcohol, the plot of $\log P_0/P_t$ against time *t* gave two intersecting linear portions, the earlier portion having a lower slope than the later. It is suggested that this may be due to interaction between the ethyl group and the methylene groups of the polymer chain in addition to the interaction between the hydroxyl group and hydrogen bonds; alternatively, it may be due to slower diffusion of the larger alcohol molecules. For fibres degraded by treatment with 3*N*-H₂SO₄ at 50°C. and relaxed in water, increased *k* values were found, and *k* also showed an increased temperature dependence.

The application of the results of these investigations to the identification and practical testing of polyamide fibres is discussed. J. C. F.

Thermal Degradation of 6,6-Nylon. I. Goodman. *J. Polymer Sci.*, 17, 587-590 (Aug. 1955).

The products of pyrolysis of dibutyladipamide, used as a model for 6,6-nylon, have been examined by ultraviolet, infrared, and mass spectrometry. 2-*n*-Butylcyclopentanone and 2,5-dibutylcyclopentanone were detected and isolated. 2-*n*-Propyl-3:4:5-bis(trimethylene)pyridine was also found. Pyrroles occur to a very small extent, and other minor products of pyrolysis are given. Direct evidence is given for the existence of analogous species in thermally degraded nylon. The close resemblance of the pyrolysis products shows that substituted adipamide decomposition is a very complex process involving fission of each bond in and adjacent to the amide linkage and the participation in synthetic processes of the α -methylene group in the amide segment. Cross-linking in 6,6-nylon involves similar reactions. A simple amide linking mechanism can play only a minor part, and a cross-linking dehydration mechanism seems to be excluded. W. R. M.

Polymerisation and Depolymerisation of ϵ -Caprolactam. I—Amide Interchange, Heat of Reaction, and Activation Energy. H. Yumoto. *Bull. Chem. Soc. Japan*, 28, 94-100 (March 1955).

An equation for polymerisation reaction rate is derived on the basis of amide interchange and verified experimentally. The heat of polymerisation is -3.8 kcal./mole (exothermic), in agreement with calorimetric values. The activation energy of depolymerisation is 12.5 kcal./mole and that of polymerisation is 8.7 kcal./mole, suggesting that ionisation is concerned in these reactions. W. R. M.

II—Effects of Diluent and Estimation of Size of "Segment". H. Yumoto. *Bull. Chem. Soc. Japan*, 28, 101-106 (March 1955).

Quantities of lactam oligomers are larger when the system is diluted. The segment, or unit of macro-Brownian motion, is estimated to be about 20 atoms. The heat of the transforming reaction between a structural unit (opened state) and a lactam monomer (closed state) is 3.4-3.6 kcal./mole. W. R. M.

Microscopical Test for Characterisation of Different Radial Shells in Perlon. O. De Riz, B. Hauptmann, and E. Schuller. *Melliand Textilber.*, 36, 680-685 (July 1955).

Carefully controlled treatment with zinc chloriodide reveals a cuticle in Perlon, the thickness of which varies with the degree of stretching. Several degrees of orientation are shown by cross-sectional photomicrography. S. R. C.

Polyvinyl Chloride Fibres (Tabulated Data). P. A. Koch. *Z. ges. Textilind.*, 57, 396-400, 403 (1955); *Textil-Rund.*, 10, 244-251 (May 1955).

Mixed-polymer Fibres (Tabulated Data). P. A. Koch. *Z. ges. Textilind.*, 57, 757-760 (20 June 1955).

PATENTS

Treating Regenerated Protein Fibres. Courtaulds, BP 734,950

A tow of regenerated protein fibres may be treated for a considerable time with a liquor of similar specific gravity by first passing the wet tow through an inert water-immiscible liquid having a suitable specific gravity so that the liquid is entrained in the fibres and keeps them floating on the treating liquor. W. G. C.

Crimped Polyester Fibres. ICI, BP 735,027

Terylene is crimped by heating it as it passes from feed rolls to draw rolls which rotate at least three times as fast as the feed rolls. The draw rolls have intermeshing teeth and the crimp is more permanent if the draw rolls are kept at a temperature not less than those to which the Terylene is heated between the rolls. C. O. C.

Stabilising the Crimp of Polyacrylonitrile Fibres. American Cyanamid, BP 735,208

The crimp of a longitudinally crimped gelled fibre containing > 35% polyacrylonitrile is stabilised by heating in steam under acid conditions. Discoloration by heat is also reduced by this treatment. W. G. C.

Improving the Heat Stability of Polyacrylonitrile.

Ameriou, Cyanamid. *BP* 735,183
The discoloration of fibres containing > 35% polyacrylonitrile is reduced by heating in water at < pH 5.8 and 50–150°C.
W. G. C.

Courpleta—The Dyeing and other Properties of Cellulose Triacetate Yarn and Staple (VIII p. 687).

Handling Blends of Dacron and Cotton (VIII p. 688).

Dyeing Polyacrylonitrile Threads or Films (VIII p. 688).

Removing Hair and Wool from Animal Hides and Skins (XII p. 693).

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Systematic Study of Sizing. VI—Effect of Various Auxiliaries on Enzymatic Desizing. R. Schutz and S. Marguer. *Bull. Inst. Text. France*, (53), 7–12 (May 1955).

The effect of wetting agents, antiseptics, and softening agents on the enzymatic removal of a starch-based size has been examined using (a) a pancreatic amylase, (b) a bacterial amylase, and (c) a malt amylase. Enzyme (a) was found to be completely inhibited by 0.01% CuSO₄. All the other products tested had no inhibitory effect. The wetting agents improved the regularity of the desizing without increasing its rate. Enzyme (b) showed a slightly retarded effect in the presence of two products based on ethylene oxide. The action of (c) was inhibited or retarded by all the wetting agents tested except two of non-ionic type; amongst the antiseptics, CuSO₄ and salicylic acid act as inhibitors, and ZnSO₄ and p-chloro-m-xylenol as retardants. One of the softening agents tested, a partly sulphonated tallow, also showed strong inhibitory action.
J. C. F.

Soiling Characteristics of Textile Fibres—I. A. S. Weatherburn and C. H. Bayley. *Text. Research J.*, 25, 549–558 (June 1955).

Methods are described for measuring the comparative soiling tendencies of textile fibres. It is shown that reflectance readings on soiled fibres measure an overall effect, part of which can be attributed to the weight and particle size distribution of the retained soil and part to the optical properties of the fibres themselves. It is suggested that the "effective soil content", which is defined as the weight of soil retained multiplied by the specific absorption (of light) of that soil, is a more significant criterion of soiling than is either the weight of soil retained or the decrease in reflectance produced by the soil. Relative values for the effective soil content can be calculated from reflectance readings by use of the Kubelka-Munk equation. The soil retention of the fibres studied increased in the following order—cotton < acetate rayon < viscose rayon < nylon (variable, depending upon soiling conditions) < wool. The same order was maintained when the time of contact with the soil varied from 5 to 80 min. The soil retention of all the fibres increased with decreasing moisture content of the yarn. The presence of even small amounts of oily material in the yarn increased substantially the amount of soil retained; on the other hand, it was apparent that the oil content of the soil had little, if any, influence on the soil retention of the yarn.
W. P. M.

Micelle Formation and Solubilisation in Benzene by Fatty Acid Salts of Higher Amines. A. Kitahara. *Bull. Chem. Soc. Japan*, 28, 234–238 (June 1955).

Micelle formation has been shown from the relationship between the van't Hoff factor and the concentration. The amount of solubilised water is proportional to the concentration of detergent. The value for the critical micelle concentration is obtained from this proportionality. With increase in the number of carbon atoms in the amine or acid part, the critical micelle concentration increases and solubilising power decreases, in contrast to results obtained with aqueous solutions.
W. R. M.

Effect of Kiering on the Crystallinity of Cotton Cellulose. M. L. Nelson, L. Segal, and H. M. Ziffle. *Text. Research J.*, 25, 534–540 (June 1955).

The effect on the crystalline-amorphous ratio of a laboratory kiering and of alcohol extraction and boiling in water under pressure has been studied. Acid-hydrolysis crystallinity determinations and moisture-sorption measurements on three varieties of cotton have indicated — (1) that standard purification treatment can increase the crystallinity of raw cotton; (2) that the extent of crystallisation varies with the individual sample; and (3) that the crystallinity values reported in the literature for purified cottons are probably higher than those for native cottons because of the changes induced by the purification treatments.
W. P. M.

Continuous Peroxide Bleaching. W. Hundt. *Textil Praxis*, 10, 474–479 (May 1955).

A review of cotton bleaching by the peroxide process including methods of continuous treatment and illustrations of the types of equipment employed.
B. K.

Modernisation of Cotton-bleaching Plants. H. Merk. *Textil Praxis*, 10, 471–473 (May 1955).

A review of bleaching processes for cotton with special reference to the value of continuous bleaching. Based on American practice, continuous bleaching of cotton is found to give an equally good bleach to that produced by normal batchwise methods even with poor qualities of cotton. The use of a short liquor ratio, viz. 1:1, as is the case with continuous processes, results in cheaper processing, although it is essential to treat large batches to obtain a saving in bleaching costs as compared with traditional batch treatments.
B. K.

Bleaching Jute with Hydrogen Peroxide. H. Chatterjee and K. B. Pal. *J.S.D.C.*, 71, 525–530 (Sept. 1955).

A single-bath process of bleaching jute with hydrogen peroxide is described with particular reference to loss of weight, lignin, and breaking strength as well as the colour of the bleached material and the composition of the residual lignin. The influence of variables—e.g. reaction temperature, time, bleach concentration, liquor ratio, pH, bleachability with peroxide compared with chlorite—has been studied.

Jute may be bleached white with peroxide with only a small loss of lignin, and the composition of its residual lignin remains more or less unchanged. It is believed that the point of attack by peroxide is a phenolic hydroxyl group and that the colour change is mainly due to an unknown structural modification of native lignin. A one-volume bath of peroxide containing a stabiliser under alkaline conditions, and a 1% solution of acidified chlorite at a liquor ratio of 10:1 and a temperature of 60–70°C., are suitable for bleaching jute. In both cases over 70% of the yarn strength can be maintained in the fully wet condition. The tendency of the bleached material to become yellow on storage is much less pronounced with peroxide than with hypochlorite or chlorite. The quality of such a yarn is unimpaired by long storage.

AUTHORS

PATENTS

Bleaching Cellulose Esters with Chlorites. Hercules Powder Co. *USP* 2,694,060

Cellulose esters of high thermal colour stability and suitable for use in high temperature moulding are prepared by bleaching them with inorganic chlorite.
C. O. C.

Inhibiting Corrosion when Bleaching Cellulose Derivatives with Chlorites. Hercules Powder Co. *USP* 2,694,059

Addition to a chlorite solution of a phosphoric acid or a derivative of such acid whose aqueous solution has pH < 7 prevents it corroding metals.
C. O. C.

Adsorption of Surface-active Agents at Solid-Liquid Interfaces (V p. 681).

Handling Blends of Dacron and Cotton (VIII p. 688).

VIII—DYEING

Relationship between Crystal Structure and Habit-modifying Dyes. J. Whetstone. *Trans. Faraday Soc.*, 51, 1142-1153 (Aug. 1955).

Correspondence between the patterns of appropriate polar groups of dye and ions of the crystal plane is necessary for dye absorption to occur on a crystal plane by electrostatic attraction. A planar dye structure is very important. The fitting of polar groups of 26 selected dyes on to ions of crystal planes of 4 inorganic oxy-salts has been tested. While the observed habit modifications are consistent with this hypothesis, the effect of many polar group orientations in increasing dye aggregation tendencies is also important in determining whether or not a dye will be a habit modifier.

W. R. M.

Explanation of Dyeing Mechanisms in Terms of Non-polar Bonding. A. N. Derbyshire and R. H. Peters. *J.S.D.C.*, 71, 530-536 (Sept. 1955).

Inadequacies are demonstrated in previous attempts to explain dyeing mechanisms on the assumption of polar forces between dye and fibre. The important part played by non-polar forces in adsorption phenomena generally is illustrated by examples. It is postulated that such forces are mainly responsible for dye-fibre attachment and that in aqueous solution they occur between hydrophobic surfaces.

The relative importance of hydrophobic and hydrophilic properties in fibres is deduced from their moisture regain figures and is discussed in relation to fibre structure. The properties of dyes are then similarly considered. Thence the dyeing behaviour of each type of fibre is predicted from the postulate that dye substantivity is due to non-polar forces. It is concluded that this assumption is sufficient to explain the methods adopted for dyeing different fibres and that it eliminates anomalies incapable of explanation on the assumption that dye-fibre attachment is due to polar forces.

AUTHORS

Substantivity of Dyes. H. Wahl. *Textil-Rund.*, 10, 413-418 (Aug. 1955).

Summary of a lecture originally published in *Teintez*, 19, 667 (1954) (abstracted in *J.S.D.C.*, 70, 590 (1954)).

Kinetics of Dyeing. J. Degarra, *Ingeniería Textil*, 22, 117-131 (March-April 1955).

A critique of work on the velocity of dyeing and the factors influencing this. The effects on equalising are referred to.

S. R. C.

Stock Dyeing. J. Bauer. *Amer. Dyestuff Rep.*, 44, P 497-P 500 (18 July 1955).**Improvement of Lustre of Cotton. XI—Dyes and Lustre.** L. Fourt, H. J. Elliott, and P. Streicher. *Text. Research J.*, 25, 326-329 (April 1955).

Lustre as measured by the contrast ratio (45°, 45° reflectance divided by 45°, 0° reflectance) tends to increase with depth of dyeing or increasing absorption of light, since this has greater effect on scattering from depth than on front surface reflection. Dyes which form particles on the surface, or show more tendency to rub, such as the lake-forming dyes which are attached to cotton by mordants, show less increase in lustre. Vat dyes which increase in crystallinity on soaping show a decrease in lustre after soaping, indicating increased scattering by larger particles.

W. F. M.

Dyeing of Textile Fibres above 100°C. J. A. Fowler. *J.S.D.C.*, 71, 443-450 (Aug. 1955).

The application is described of high-temperature techniques, i.e. above 100°C., in the dyeing of a variety of textile fibres. Particular mention is made of the application of direct and vat dyes to cellulosic fibres, of acid and chrome dyes to wool, and of disperse and azoic dyes to Terylene polyester fibre. Reference is also made to the machinery available and the advantages to be gained by the use of high-temperature techniques.

AUTHORS

Dyeing of Viscose Rayon. A. Wehrung. *Textil-Rund.*, 10, 359-364 (July); 428-432 (Aug. 1955).

A review of theories and experimental work relating to the dyeing of viscose rayon, and the influence of the physical properties of the fibre on dyeing behaviour.

J. C. F.

Chemistry of Esters of Leuco Vat Dyes—I. S. Ainsworth and A. Johnson. *J.S.D.C.*, 71, 592-604 (Oct. 1955).

The oxidation with acidic hydrogen peroxide of the disulphuric esters of leuco anthraquinone-2-sulphonic acid and of leuco indigo-5:7:5':7'-tetrasulphonic acid has been investigated. It has been shown that direct oxidation of the ester occurs and is mainly a univalent change, in which only one sulphate group is eliminated from the ester, leading to the formation of a relatively stable semiquinone derivative. Oxidation with nitrous acid has been examined briefly, and it has been shown that oxygen plays an important part.

AUTHORS

Phototropy of Indanthren Dyes. H. Waibel. *Melliand Textilber.*, 36, 737-740 (July 1955).

Tabular information is given on untreated dyeings and the effect of depth of dyeing on the phenomenon. The influences of the fibre, the finishes, and the presence of other dyes are considered.

S. R. C.

Modification of and Possible Damage to Wool in Dyeing. A. Würz. *Melliand Textilber.*, 36, 580-597 (June); 810-813 (Aug. 1955).

Dyeing metal-complex dyes with 10% H₂SO₄ causes markedly more damage than 6% H₂SO₄ + auxiliary. Such processes are suitable more for heavy materials such as gabardine, whilst the normal acid dyes or the newer metal-complex dyes applied weakly acid are more suitable for weaker structures.

S. R. C.

Influence of pH of Dye bath on Wool Quality. E. Schönpflug. *Textil Praxis*, 10, 490-493 (May 1955).

The effect of variations in dyeing processes, especially changes in pH, on wool quality is studied by examining the following—alkali solubility, acid solubility, cystine content, and tensile strength.

B. K.

Some Observations on the Dyeing of Wool at High Temperatures. G. A. Coutie, D. R. Lemm, and H. Sagar. *J.S.D.C.*, 71, 433-443 (Aug. 1955).

It has been shown that dyeing temperatures above 100°C. can result in the decomposition of a number of wool dyes, primarily as a result of reduction. The full wet fastness properties of wool dyes can be obtained in short times at elevated temperatures. Quantitative tests indicate marked increases in the rate of redistribution of dye at elevated temperatures. Tests on the physical properties of worsted yarns show that excessive degradation of the wool fibre occurs at temperatures above the boil if strongly acid or neutral dye baths are employed. It has been shown that wet tenacity is a much more sensitive criterion of fibre degradation than dry tenacity. Conditions of time, temperature, and pH are suggested which will yield satisfactory dyeings at elevated temperatures without producing undue fibre degradation.

AUTHORS

Dyeing of Acetate Rayon with Disperse Dyes. IV—Adsorption Isotherms. C. L. Bird and F. Manchester. *J.S.D.C.*, 71, 604-609 (Oct. 1955).

Equilibrium isotherms have been obtained at 80°C. with acetate rayon and five disperse dyes. Two azo dyes showed a linear relationship up to saturation between dye on the fibre and dye in the aqueous phase. Non-linear curves were obtained with three anthraquinonoid dyes, but a straight line was obtained when a solubilized 1-methylamino-4-anilinoanthraquinone was used in place of a dispersion. With purified Dispersol Fast Scarlet B the slope of the isotherm was found to be independent of liquor : yarn ratio and inversely proportional to the concentration of dispersing agent over a limited range.

The adsorption by acetate rayon of purified Limapols C and LS has been studied at 80°C. Both detergents hydrolyse gradually in hot aqueous solution.

AUTHORS

Courplets—The Dyeing and other Properties of Cellulose Triacetate Yarn and Staple. J. Boulton. *J.S.D.C.*, 71, 451-464 (Aug. 1955).

The manufacture of Courplets is briefly described, and a short historical review given of the development of cellulose triacetate fibres. Detailed physical and chemical properties of the fibre are enumerated. In overall behaviour as a textile fibre, Courplets closely resembles

the synthetic fibre. On the other hand, its dyeing properties are more nearly like those of secondary acetate. This spread of properties, with especial emphasis on the ready dyeability of the fibre in contrast to synthetics, and its capacity to provide durable pleating and embossing effects, establish it, in view of its price, as a fibre of great promise. The techniques of dyeing so far developed are discussed in detail, and a description is given of durable pleating effects. Tables of uses and colour fastnesses are provided.

AUTHOR

Dyeing, Finishing, and Heat-treating Arnel Cellulose Triacetate. F. Fortess. *Amer. Dyestuff Rep.*, 44, P 524-P 537 (1 Aug. 1955).

Arnel (Celanese Corp. of America) cellulose triacetate can be dyed with disperse dyes at temp. close to the boil without saponification or delustring. The dyeing rate is slower than on secondary acetate rayon; dyeing on closed jigs or winches is possible in normal times for light and medium depths, and accelerators, e.g. 10% tripropyl phosphate, permit heavy dyeings. Data are presented on the effect of accelerators on dyeing rate. Comprehensive information is given on preparation and finishing of Arnel fabrics, with a table of selected dyes. Dyeing and finishing of blends containing Arnel are also discussed, though more briefly.

J. W. B.

Dyeing Modern Synthetic Fibres. J. Nusslein. *Z. ges. Textilind.*, 57, 900-906 (20 July 1955).

A review of synthetic fibres including chemical structure, properties, dyeing, and application in the textile industry.

B. K.

Dyeing and Finishing Synthetic Tubular Knit Goods. R. C. Geering. *Amer. Dyestuff Rep.*, 44, P 459-P 463 (4 July 1955).

A flow-sheet of processes in finishing synthetic tubular knitwear is presented, and the principles and equipment are discussed.

J. W. B.

New Developments in Tricot Dyeing. L. L. Walmsley. *Amer. Dyestuff Rep.*, 44, P 490-P 493 (18 July 1955).

Problems concerning the production of nylon tricot fitted bed sheets are discussed with reference to choice of fibres, yarn sizes, setting procedures, and cutting methods.

J. W. B.

Handling Blends of Dacron and Cotton. J. S. Rumsey. *Canadian Textile J.*, 72, 41-44 (29 July 1955).

Data are given concerning the processing of Dacron-cotton blend fabrics. Desizing, scouring and bleaching, control of "pilling", union and cross dyeing, and finishing operations are described. It is considered that the blend must contain 65-70% of Dacron to achieve satisfactory wash-and-wear performance.

A. H.

Dyeing of Terylene Polyester Fibre with Disperse Dyes above 100°C. A. S. Fern. *J.S.D.C.*, 71, 502-513 (Sept. 1955).

The stability, dyeing, and fastness properties of a range of disperse dyes have been studied on Terylene polyester fibre at temperatures up to 120°C. under laboratory conditions. The potential advantages and disadvantages of dyeing under such conditions on the large scale are pointed out.

AUTHOR

Acrylonitrile Copolymers and their Dyeing Properties. G. E. Ham. *Z. ges. Textilind.*, 57, 594-597 (5 June 1955).

The various organic derivatives of acrylonitrile are described with particular reference to the dyeing properties of the copolymers.

B. K.

Irreversible Photobleaching of Solutions of Fluorescent Dyes. I—Kinetic Studies on the Primary Process. M. Imamura and M. Koizumi. *Bull. Chem. Soc. Japan*, 28, 117-124 (March 1955).

The rate of photobleaching of eosin depends only on, and is proportional to, the quantity of absorbed light for a dye concn. of 10^{-4} mole/litre. The overall rate constant and the quantum yield do not change over a wide range of oxygen concn., but begin to decrease below a certain amount. An expression is given for the rate constant for alcohol-water mixtures. Possible mechanisms of photobleaching of eosin by oxygen are discussed. W. R. M.

Dyeing Suède Leather—I. K. Eitel. *Leder*, 5, 290-3 (1954); *Chem. Abs.*, 49, 7859 (10 June 1955).

As the Cr_2O_3 content of the leather increased from 4-14%, absorption of dye increased up to a maximum at 8% Cr_2O_3 and then decreased.

C. O. C.

Dyeing Leather with Anthrasol Dyes. *Leder*, 6, 5-9 (1955); *Chem. Abs.*, 49, 8624 (25 June 1955).

Anthrasol dyes can be applied to leather in three ways: (1) Dye with NaNO_2 in the liquor and then treat with H_2SO_4 ; (2) Treat the dyed leather with H_2SO_4 and $\text{K}_2\text{Cr}_2\text{O}_7$ and remove the chrome colour with H-COOH-NaHSO_4 ; (3) Dye with tartaric acid and aluminium chloride in the bath and then irradiate with a strong arc lamp. Anthrasol Blue IBC, which is a tetraester, may become yellowish if overoxidised because it loses two imino H atoms, but this can be prevented by addition of urea.

C. O. C.

PATENTS

Improving the Wet and Light Fastnesses of Dyeings and Prints made with Any Water-soluble Substantive Dye on Any Fibre. General Aniline. BP 735,619

Treatment with an aqueous solution of a composition comprising a copper compound, an amino carboxylic acid and an acidic condensate of formaldehyde, diacyandiamide and an ionisable ammonium salt, improves the fastness to light and water of dyeings and prints made with any water-soluble substantive dye on any fibre. The compositions are substantive and exhaust well.

C. O. C.

Improving the Wet Fastness of Direct Dyes on Cellulose. BASF. BP 735,557

Treatment of direct dyeings on cellulose with vinyl polymers containing basic N groups and free from cyclic carbonyl groups, e.g. N-vinylimidazol polymers, or their water-soluble salts or quaternary ammonium compounds, improves their wet fastness. The effect is increased if the dyeings are treated before, simultaneously with or after the above treatment, with water-soluble salts of Cu, Co, or Ni.

C. O. C.

Dyeing Polyacrylonitrile Threads or Films. Société Rhodacéta. BP 734,974

The material is treated with a dye or compound giving rise to a dye between starting to wet-draw the material and the first drying after that drawing. This enables dyes to be used for which the material has normally no affinity. Thus a polyacrylonitrile filament is drawn in water at 95°C., 50 cm. away from this bath the filament passes at ordinary temperature into a rotating drum immersed in an aqueous soln. of 2 g./litre of C.I. 671 and is then dried. The resulting dyeing is fast to boiling in 1% soap soln. for 1 hr. A filament drawn and dried without passing through this dyebath shows no affinity for the dye when immersed in the same dyebath.

C. O. C.

Vat Dyeing of Nylon. General Aniline. USP 2,693,994

Treatment of nylon with a 1.0-2.75% (by volume) aqueous solution of a 2-5% solution by weight of phthalic anhydride in a phenol at 150-210°F. for 2-20 min. enables it to be dyed deeply with vat dyes to give dyeings of improved fastness to light.

C. O. C.

Comments on Package-dyeing Machines (I p. 670).

Experimental Application of a Turbodynamic Principle in Dyeing (I p. 670).

Reaction between Rhodamine B and Antimony (IV p. 674).

Concentrated Stable Solutions of Reduced Indophenol Sulphurised Vat Dyes (IV p. 680).

IX—PRINTING

Printing of Wool, including Mélange or Vigoureux Printing of Other Staple Fibres. J. A. Potter. *J.S.D.C.*, 71, 645-652 (Nov. 1955).

The principles of textile printing are outlined, as a background to a discussion of the preparation and the composition of printing pastes for application to wool cloth and the selection of dyes for this purpose. The technique of printing wool in the manner originated by Vigoureux is also described, and the selection of dyes and the composition of the printing pastes are discussed. The extension of this technique to recently developed synthetic fibres and to fibre mixtures is considered.

AUTHOR

Flock Printing. W. Pistorius. *Z. ges. Textilind.*, 57, 777-780 (20 June 1955).

A survey of flock printing of textile materials with special reference to the binders used and the properties of an ideal binder. B. K.

PATENTS

Use of Concentrated Colour Pastes in Long Runs. Rock Hill Printing & Finishing Co. *USP* 2,691,602

Addition of an organic solvent soluble water repellent, particularly a polysiloxane of formula $(R_2SiO)_xR$ (R = hydrocarbon radical; y = 1-3; x = a whole number), e.g. Antifoam A (Dow Corning), to a concentrated colour paste prevents the paste drying on the non-etched surface of the intaglio roller. This enables concentrated colours to be used during long runs without the pigment drying on the non-etched surface of the roller thus preventing both scratching of the roller and marking of those portions of the print which should be left blank. C. O. C.

Printing or Decorating Ceramics, Glass or Plastics. G. L. Murray. *BP* 735,637

A solid resilient pad having a portion of generally parabolic shape is pressed against the surface of an engraved, litho or etched plate to remove the colour therefrom. It is then pressed against the curved surface of the article to be printed by first contacting that surface at the centre and then accurately developing the design outwardly from the point of first contact over the surface by a generating action with a progressively increasing boundary line contact. C. O. C.

Colour Correcting in Photochemical Processes. Colour Separation Techniques. *USP* 2,692,825

A colour correcting method usable in various types of printing but particularly in photogravure printing. C. O. C.

Colour Photography. S. Papadakis. *BP* 734,932

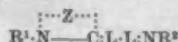
A suitable support is treated with the sulphuric ester of the leuco compound of a blue dye which is oxidised by light, e.g. sulphuric ester of leuco 5:7:5':7'-tetrabromo-indigo, exposed to light through the negative obtained by using an orange screen and the resultant image developed. This procedure is repeated using a red and yellow dye, e.g. sulphuric esters of leuco 1:2-antraquinone acridone and flavanthrene, using the green and violet screen negatives respectively. C. O. C.

Light-sensitive Diazotype Layers containing Magnesium Salts. General Aniline. *USP* 2,694,010

Brighter images and whiter backgrounds with resulting improved visual contrast are obtained if the zinc chloride in the diazo material is replaced by a mixture of 90-60% $ZnCl_2$ and 10-40% of a water-soluble inorganic magnesium salt, e.g. $MgCl_2$ or $MgSO_4$. C. O. C.

Supersensitisation of Carbocyanine Dyes with Hemicyanine Bases. Eastman Kodak Co. *USP* 2,694,638

Compounds of formula—



(R^1 = subst. or unsubst. Alk; L = subst. or unsubst. methenyl; R^2 = Ar; Z = atoms to complete a heterocyclic β -naphthothiazoline nucleus), e.g. 1-ethyl-2-(2-phenyliminopropylidene)- β -naphthothiazoline, render certain carbocyanine dyes, e.g. 2:2'-carbocyanine, 9-alkylthia-2'-carbocyanine and some *meso*-subst. carbocyanine dyes, much more sensitive to light. C. O. C.

Colour Couplers (IV p. 677).

Improving the Wet and Light Fastnesses of Dyeings and Prints made with Any Water-soluble Substantive Dye on Any Fibre (VIII p. 688).

X—SIZING AND FINISHING

Decimal Classification—Applicability to the Textile Finishing Industry. E. Cucho. *Textil-Rund.*, 10, 374-379 (July 1955).

The author discusses the definitions of the comprehensive terms relating to textile finishing processes, and puts forward a plea for the modification of that part of the International Decimal Classification which deals with this subject. J. C. F.

Some Scientific Aspects of the Finishing of Wool Fabrics. E. G. Carter. *Bull. Inst. Text. France*, (53), 13-31 (May 1955).

The rôle of the finisher in playing upon the special physical and chemical properties of the wool fibre in order to draw out all that is possible in the way of texture, handle, and warmth in the finished fabrics is discussed. Felting plays an important part in finishing processes, and understanding of it is facilitated by modern theories. The scientific study of raising and setting may be expected to lead to more permanently stable textures. The surface deposition of high polymers including rubber and nylon on wool fabrics offers possibilities of increased resistance to wear and washing shrinkage. J. C. F.

Potential Applications of Silicones in Textile Finishing. M. Kramer. *Melliand Textilber.*, 36, 806-809 (Aug. 1955).

Waterproofing, coating, and foam prevention are discussed. S. R. C.

Effect of Colloidal Silica Treatments on Cotton Processing Characteristics and Yarn Quality. H. M. Brown, J. H. Langston, and W. T. Rainey. *Text. Research J.*, 25, 462-471 (May 1955).

Preparations of colloidal silica and quartz increase the interfibre friction of cotton fibres. From measurements of interfibre friction forces of treated fibres it was found that, in general, compounds of silica and titanium dioxide caused the greatest increase in frictional force. Several cottons have been processed, and definite improvements in processing result from the use of selected additives to increase the frictional characteristics of the fibre. Stronger, more uniform yarns, which are easier to spin, can be produced. The improvement in spinning is greatest for the cotton of lowest staple length and maturity. Thus frictional agents may allow the processing of cottons of shorter staple, which is not possible without these agents. Significantly different amounts of frictional agent are required to produce maximum drafting force with different cottons. W. P. M.

Reaction of Formaldehyde with Cellulose. R. Steele. *Text. Research J.*, 25, 545-548 (June 1955).

The acid-catalysed reaction of formaldehyde with cellulose was investigated by observing the infrared spectra of—(1) untreated cellulose, (2) cellulose treated with formaldehyde in presence of boric acid, (3) cellulose treated as in (2) and washed in boiling water for 30 min., and (4) cellulose treated as in (2) and then treated with boiling 0.1N-HCl. The results show that the number of hydroxyl groups in the product is less than in the original cellulose, so that cross-linking must have occurred; further, the cross-links most frequently occurring are methylene groups, rather than polyoxymethylene chains. Also, unbound formaldehyde present on the cellulose after the reaction, which can be removed by boiling water, is either molecularly adsorbed formaldehyde or some uncommon polymer of formaldehyde containing free aldehyde groups. W. P. M.

Reaction of Cotton with β -Propiolactone in the Presence of Alkali. G. C. Daul, R. M. Reinhardt, and J. D. Reid. *Text. Research J.*, 25, 330-333 (April 1955).

The reaction of cotton cellulose with β -propiolactone may be effected in the presence of strong alkalis such as sodium hydroxide. Monoethanolamine and trimethylbenzylammonium hydroxide also will catalyse the reaction, but to a lesser degree. Di- and tri-ethanolamine are ineffective. By-products of the reaction are water-soluble and may be removed by a hot-water wash. At room temperature the reaction occurs immediately upon contact of alkali-treated cellulose with β -propiolactone and is essentially complete in 5 min. At lower temperatures the reaction is slower but more easily controlled and there are less undesirable by-products. Maximum reaction occurs with a concn. of 15-20% NaOH. Diluents such as xylene and *tert.*-butyl alcohol, which react with the lactone only with difficulty, are the most efficient. The products are mixed ethers and esters of cellulose and polyesters thereof, in fibrous form. The ether linkages are stable to saponification, while the ester linkages are broken. Therefore saponification removes all but the carboxyethyl groups attached directly to the cellulose molecule. W. P. M.

Treatment of Cotton Cellulose with Aqueous Solutions of Ethylamine. L. Loeb and L. Segal. *Text. Research J.*, 25, 516-519 (June 1955).

Aqueous solutions of ethylamine (70-100%) have been investigated as to their effectiveness in reducing the crystallinity of cotton cellulose. The degree of crystallinity of cotton cellulose was found to be little affected by ethylamine solutions containing 70% of the amine. The amine solutions of slightly higher concentration strongly reduced both the crystallinity and the levelling-off D.P. This reduction continued with further increase in amine concentration. The sharp break in effectiveness at 70% ethylamine is discussed in terms of hydrogen-bonding capacity in the amine-water-cellulose system, and it is suggested that it is due to the inability of the more dilute solutions of the reagent to form the amine-cellulose complexes prerequisite to decrystallisation. W. P. M.

Effects of Chemical Attack on the Physiological Properties of Textiles. O. Mochoels. *Melliand Textilber.*, 36, 722-728 (July 1955).

The influences of chemical damage on the insulating properties, water uptake, perspiration transfer, and the effect of micro-organisms are reviewed. Wool treated with $K_2Cr_2O_7$ has modified resistance to bacteria; linen peroxide-bleached and scoured varies in water uptake; the waterproofing of poplin affects the heat insulation of the fabric; and the chromium content of treated Perlon influences the water uptake. Wet finishing should therefore be carried out with the end-use in mind. S. R. C.

Some Chemically Modified Celluloses and their Resistance to Fungal Degradation. R. Thomas. *Text. Research J.*, 25, 559-562 (June 1955).

Cellulose has been chemically modified by partial oxidation with periodic acid followed by coupling with various carbonyl reagents. The modified celluloses have been tested for resistance to fungal degradation, and some have been shown to inhibit growth at relatively low degrees of substitution (D.S.). There is no obvious correlation between the amount of protection conferred and either the chemical nature of the substituent group or the D.S. The most effective substituent tested was phenylhydrazine, which confers complete resistance to the growth of *Memnoniella echinata* at a D.S. of 0.37%. Some aspects of the inhibitory mechanism and the suitability of the method as a fungus-proofing procedure are discussed. W. P. M.

Protection of Textiles from Chemical Attack and Mildew. H. M. Ulrich. *Textil Praxis*, 10, 278-281 (March); 366-378 (April 1955).

A review of products which inhibit fibre damage, including natural tannins and organic, metallic, and organometallic compounds. Antiseptic compounds for sizes, finishes, adhesives, thickeners, etc. are listed, and the specifications for a good antiseptic for textile usage are given—economical in use, readily soluble or emulsifiable; resistant to heat (singeing, drying, calendaring); not liable to attack metals (rollers, etc.); no effect on sizes, finishes, and fibres; non-volatile; not liable to form coloured compounds or to stain fibres; free from smell; no action on enzymes; and no irritant action on the skin.

Details are given regarding the behaviour of antiseptic opd. on the fibre applied in size or finishing agents, quantities of various antiseptic materials likely to be present on the fibre, the solubility of antiseptic agents in water, caustic soda, and organic solvents, and detailed methods for their identification. B. K.

Permanent Protection of Textiles against Microbiological Attack. J. Salquin. *Tintex*, 20, 609-627 (Aug. 1955).

The types of micro-organisms responsible and their mode of action on animal and vegetable fibres and on leather are reviewed. The protective methods considered include creation of an impenetrable envelope round the fibre, modification of physical structure, use of inert fibres, and modification of chemical structure by acetylation, phosphorylation, methylation, cyanoethylation, and carboxymethylation. The use of waterproofing agents or destruction of the micro-organisms by means of phenolic compounds, mercurials, contact insecticides, copper complexes, zinc salts, quaternary ammonium compounds, chrome salts and tannages, and salicylic acid derivatives are dealt with in conclusion. S. R. C.

Technology of Shrink- and Crease-resistance. F. Weber. *Textil-Rund.*, 10, 226-235 (May 1955).

Resin finishes designed to impart shrink- and crease-resistance to cellulosic fibres are discussed with particular reference to the effect of such finishes on the general properties of the fibre. A tabular analysis is given of 92 patented resin treatments under the headings—(1) year of patent, (2) patentee, (3) patent number(s), (4) type of resin and additives, (5) degree of dispersion, (6) catalyst, (7) hardening temperature, (8) additional remarks. J. C. F.

Principles and Practice of the Flameproofing of Textiles. F. Ward. *J.S.D.C.*, 71, 569-578 (Oct. 1955).

The flameproofing of textile fibres and fabrics is considered in all its aspects.

Flammability and associated properties are defined. The methods at present available for rendering cellulosic fibres flameproof are described—soluble finishes, the so-called "permanent" ester finishes, precipitation treatments, and coatings. The flameproofing of cellulose acetate, wool, nylon, and blends is also discussed.

A new theory is suggested for the basic mechanism of the burning and the flameproofing of fabrics. The methods of testing flammability and related properties are critically described, and the difficulty of interpreting laboratory data in terms of actual fire hazard is shown.

Some new data on the ignition temperatures and the vertical burning rates of a series of fibres are given.

AUTHOR

Effect of Flameproofing Agents on Cotton Cellulose.

H. A. Schuyten, J. W. Weaver, and J. D. Reid. *Ind. Eng. Chem.*, 47, 1433-1439 (July 1955).

Flameproofing agents are shown to act as catalysts, accelerating the rate of decomposition and decreasing gas production, on ignition of cotton. Although the gases produced are flammable, they are evolved at a much lower temperature than in the case of untreated cotton, and are dissipated at temperatures too low for ignition. The reaction involved in flameproofing is dehydration, and analyses of the char correspond with 80% theoretical dehydration of cellulose. In all cases examined, Lewis acids, which may be formed only on the thermal decomposition of the applied flameproofing agent, are the effective flameproofing agents, and these are responsible for the catalytic dehydration of the cellulose. The inefficiency of the flameproofing treatments on cellulose acetate is explained by the production, by the action of the acids, of flammable acetic acid in place of water. W. K. R.

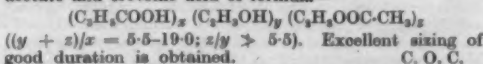
Cellulose Acetate and Water Repellency. R. J. Mann. *J.S.D.C.*, 71, 579-587 (Oct. 1955).

The methods in use for making fabric water-repellent and the methods of measuring these properties are briefly reviewed. Experiments are described to determine the best available method for increasing the water-repellency of cellulose acetate, special attention being given to the silicones on account of their superior properties and comparative unfamiliarity. AUTHOR

PATENTS

Sizing Nylon Yarn. DuP. BP 735,358

Nylon is sized with a hydrolyzed copolymer of vinyl acetate and crotonic acid of formula—



Non-slip Finish. Monsanto. USP 2,693,427

Excellent slip resistance and, in the case of knitted materials, resistance to laddering is obtained without loss of handle by treatment first with a cationic surface active agent and then with a dilute solution of colloidal silica. C. O. C.

Permanent Waving of Human Hair. Gillette Co. BP 735,303

It is unnecessary for breaking of the disulphide bonds and development of the wave by plastic flow to be simultaneous. Thus the wound hair is treated with the reducing agent long enough to break the disulphide bonds and soften the hair but insufficient to fully develop the wave. The hair is then rinsed with water and the diluted reducing

soln. kept on the wound hair long enough to obtain complete air oxidation of the hair and then the hair is unwound. C. O. C.

Durable, Stiff Finish for Nylon. Rohm & Haas Co. USP 2,692,182

A crisp, stiff durable finish is imparted to nylon textiles by treating them with an aqueous solution containing polyacrylic acid (preferably one whose 5% aq. soln. at 100°r. has viscosity 2-12 centistokes) and a polyhydric alcohol, e.g. ethylene or diethylene glycol, drying, and baking at 300-450°r. The resultant finish is fast to washing. Applied before dyeing the finish acts as a mordant for basic and some disperse dyes. C. O. C.

Improving the Affinity for Dyes and Resistance to Shrinkage of Terylene. ICI. BP 735,171

Heating Terylene at 210-220°C. for a brief period renders it resistant to shrinking and enables it to be dyed deeper with disperse dyes. C. O. C.

Resiliently-bonded Fibrous Materials. Xetal Products. BP 734,594

A mass of loose hair, etc. is bonded by depositing on it, especially at the crossing points of individual fibres, a non-rubbery product obtained from a polyester and an organic polyisocyanate or polyisothiocyanate which contains iso- or isothiocyanate radicals available for reaction either immediately or upon heating. The reaction between the polyester and the iso- or isothiocyanate is then allowed or caused to proceed until a rubbery product is obtained. C. O. C.

Felting in the Bumping Stocks (I p. 670).

Imparting Permanent Fragrance to Fabrics (III p. 672).

Degradation of Viscose Rayon Textiles by Light (VI p. 682).

Iodination of Wool (VI p. 684).

Estimation of Alkali Solubility of Silk Fibroin (VI p. 684).

Improving the Quality of Silk by Chemical Treatment.

II—Treatment with Paraformaldehyde (VI p. 684).

Couplets—The Dyeing and other Properties of Cellulose

Triacetate Yarn and Staple (VIII p. 687).

Dyeing, Finishing, and Heat-treating Arnel Cellulose

Triacetate (VIII p. 688).

Dyeing and Finishing, Synthetic Tubular Knit Goods

(VIII p. 688).

Handling Blends of Dacron and Cotton (VIII p. 688).

Wear Properties of Resin-finished Rayon Staple Fabrics—

A New Method of Laboratory Assessment (XIV p. 696).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Adsorption of Carboxymethylcellulose on Cotton.

J. Stavitz, W. Klaus, and H. Krämer. *Kolloid-Z.*, 142, 166 (July 1955).

The adsorption of carboxymethylcellulose, containing radioactive carbon, from aqueous solutions on cotton has been studied. The concentration of carboxymethylcellulose in solution was not, apparently, decreased, and it is concluded that even the formation of a monomolecular film on the cotton does not occur. W. R. M.

Viscosity Behaviour of Dilute Solutions of Cellulose in Cuprammonium. M. Rigbi. *J. Polymer Sci.*, 17, 583-586 (Aug. 1955).

Plots of $\log(\eta_{sp}/c)$ against c for solutions of bacterial cellulose in cuprammonium show a steep slope and a sharp break in linearity with a minimum at concentrations below 0.03 g./100 ml. This anomaly is time-dependent, but the steep slope is permanent. Similar results are obtained with a beechwood cellulose, but the slope is steeper. Cotton treated with formaldehyde also shows a steep slope and a "minimum" anomaly. It is concluded that the steep slope is due to cross-linking, and it is inferred that beechwood cellulose is cross-linked. The minimum anomaly seems to involve a non-equilibrium state of the polymer with time effects that may be enhanced by cross-linking. W. R. M.

Structural Fragments in High-polymer Cellulose Solutions. H. Dolmetsch. *Melliand Textilber.*, 36, 766-771 (Aug. 1955).

When precipitating somewhat dilute solutions of highly polymeric cellulose, coarse filamentous aggregates appear, which can be resolved into granular bundles of uniform size. The same intermediate stage is reached by either solution or precipitation, being elements of the fibre. Their existence in solution is to be reckoned with, although the highly solvated state prevents their being observed or sedimented. The form and the shape of the elements are independent of the solvent or the source of the cellulose. S. R. C.

Quantitative X-Ray Determination of Amorphous Phase in Wood Pulp as related to Physical and Chemical Properties. G. L. Clark and H. C. Terford. *Anal. Chem.*, 27, 888-895 (June 1955).

The percentage of amorphous phase of cellulose in wood pulps has been determined by X-ray diffraction, with a technique involving quant. calibration and scattering correction standards, based upon the ratio of the (002) peak intensity of cryst. cellulose to that of the halo at 19° for amorphous cellulose. For each pulp, the absolute density was measured. Although crystallinity values for individual pulps have never been reported, the results of this work were of the same order of magnitude as the single value (~70% amorphous phase 30%) reported by workers using chemical methods. Groundwood pulps have higher amorphous contents than fibres pulped from chips, indicating that mechanical grinding degrades the cryst. regions. Bleaching increases the crystallinity, probably by removal of amorphous encrustations. The tensile strength of paper sheeted from pulp slurries appears to be a function of the amorphous cellulose content of the fibres. There is no statistical correlation between tear strength of paper and crystallinity of pulp. The density of wood pulp increases with crystalline contents up to ca. 70%, then remains almost constant. Tear strength is a function of pulp properties and processing. From X-ray diffraction analysis and density measurements, it is possible to classify wood pulps in terms of amorphous phase content, and control tensile strength, water-holding capacity, and other practical properties of paper sheets, from the pulp slurries. J. W. D.

Acetylation of Holocellulose in the Fibrous State.

T. Fujimura. *Bull. Chem. Soc. Japan*, 28, 248-253 (June 1955).

Acetylation of air-dried holocellulose is roughly controlled by surface resistance, regardless of various ratios depending on conditions of acetylation and degree of refining. The rate of acetylation of alkali-refined samples seems to be determined by internal diffusion. Birefringence decreases at first, then increases, and finally decreases again. A final higher birefringence is ascribed to acetylation. Amorphous material is initially acetylated and lost, so that residual crystallinity and birefringence increase. Alkali refining retards acetylation. There is an optimum concentration of refining alkali, which is possibly related to homification, loss of amorphous material, change of crystal form, and hindrance by residual pentosan. Pentosan in the product is not, apparently, reduced. W. R. M.

Tetra-O-methyl-D-glucopyranose from O-Methylcellulose. (a) D. I. McGilvray; (b) E. Paoou and L. Rebenfeld. *Text. Research J.*, 25, 568-569 (June 1955).

(a) Contrary to the experience of Rebenfeld and Paoou (*Text. Research J.*, 24, 941 (1954)), McGilvray states that hydrolysis of the O-methylcelluloses gives tetra-O-methyl-D-glucopyranose, which has been isolated from the hydrolysate, identified, and determined (McGilvray, *J.C.S.*, 2577 (1953)).

(b) Paoou and Rebenfeld suggest that the discrepancy between the two sets of results might be due to McGilvray's method for purification of the raw material, as a result of which the tetramethylglucoside found might have originated from impurities embedded in his "natural cellulose". They suggest also that the agreement claimed by McGilvray between the values for the D.P. determined osmotically and by end-group analysis would constitute

a powerful argument in favour of the theory of periodically occurring weak bonds in the cellulose structure.

W. P. M.

Countercurrent Fractionation of Cellulose Acetate by Selective Diffusion into Charcoal. H. A. Swenson. *Svensk Papperstidning*, 58, 550-553 (15 Aug. 1955).

Fifty fractions isolated from secondary acetates had weighted average molecular weights of ca. 35,000 and 63,000. A steady increase in intrinsic viscosity with fraction number was shown, as well as the dependence of polymer uptake by charcoal on mol. wt. and time, and the existence of a mol. wt. gradient in the charcoal. The adsorption capacity and D.P. selectivity of the charcoal can be altered by changing the polarity of the polymer solvent.

S. V. S.

Manufacture of Industrial Cellulose Nitrate. W. A. Caldwell and J. J. Creasy. *J. Oil & Col. Chem. Assoc.*, 38, 431-454 (Aug. 1955).

The theory and practice of nitration by "mixed acid" are discussed, and the relationship between N content and acid strength is indicated. The displacement and mechanical nitration processes are described, and the theory and practice of stabilisation by boiling are discussed. Stabilisation is achieved also by kieren (though it is carried out principally to effect controlled viscosity reduction), and both continuous and batch kiers are described. The manufacture of dense cellulose nitrate from wood pulp is described. Dehydration of the water-wet cellulose nitrate is carried out with various alcohols, and the "spirit strength" (dilution ratio) is determined by the hydrocarbon method, whilst the quality of the product is examined by means of critical test solvents. There are several photographs and diagrams of plant and apparatus.

J. W. D.

Degree of Polymerisation and its Distribution in Cellulose Rayons. III—Determination of the Intrinsic Viscosity of Cellulose Nitrate from a Single Measurement of Relative Viscosity.

IV—Effect of Non-Newtonian Flow on the Intrinsic Viscosity of Cellulose Nitrate. V—Fractionation of Nitrated Rayons. W. G. Harland. *J. Textile Inst.*, 46, T 464-T 471; T 472-T 482; T 483-T 499 (July 1955).

III—The relation between the relative viscosity η_r and the concentration C of solutions of fractionated cellulose nitrates in dil. *n*-butyl acetate is expressed by the equation—

$$\log_{10} \frac{\eta_r - 1}{C} = \log_{10} i + aC + bC^2 + cC^3$$

where a , b , and c are constants and i is the intrinsic viscosity. The constants are evaluated, and a table is presented of corresponding values of η_r and $\log iC$ for a concn. of 0.2 g./dl. This permits the intrinsic viscosity of a cellulose nitrate to be computed from a single measurement of relative viscosity made at about this concn.

IV—A viscometer is described whereby pressures below 10 cm. of water can be maintained sufficiently constant to obtain accurate times of flow, and the viscosities of *n*-butyl acetate soln. of a cellulose nitrate fraction of D.P. about 1700 and of a nitrated raw cotton are measured at several mean heads above and below those operating in a conventional capillary viscometer. Intrinsic viscosities are calculated from relative viscosities as they were measured at constant mean head, from relative viscosities interpolated at constant mean rate of flow, and from relative viscosities extrapolated to zero head or zero rate of flow. The equation—

$$\log \frac{\eta_r - 1}{C} = \log i + kC$$

where k is a constant, is used to determine intrinsic viscosity. At higher rates of flow intrinsic viscosity can be computed from relative viscosity at two or three concentrations only. Solutions of nitrated cotton are much more anomalous than those of the fraction, particularly at low mean heads or low mean rates of flow, and the difference extends to the intrinsic viscosity. Comparisons with earlier results are made and discussed, and it is considered that the previous inference that the rate of shear is more fundamental than the shear stress is

illogical; the use in the present experiments of viscosities at constant mean rate of shear has not simplified the difficulties of eliminating shear effects from intrinsic viscosities.

V—Four commercial rayons are fully nitrated and each product is fractionated three times by precipitation from acetone solution with *n*-hexane. Intrinsic viscosities of all fractions from the second and third stages are determined in *n*-butyl acetate and converted to D.P., and the N contents of the final fractions are measured. Satisfactory agreement is obtained from comparing values for average D.P., N contents, and weights of unfractionated nitrated rayons with corresponding values summed from the fractions. The presence of some hemicellulose is inferred from the low N contents of a few low-mol. wt. fractions. Examination of distribution curves for the second and third fractionation stages indicates that a true distribution picture cannot be obtained from a single coarse fractionation if material of D.P. > approx. 800 is present, and also that inflections in the internal distribution curve are not indicative of excess amounts of certain D.P. values resulting from weak bonds or crystallite size. The inflections are probably due to incomplete separation of the D.P.s in the last subfractions precipitated from material undergoing refractionation, and appear to be quite spurious.

J. W. B.

PATENTS

Stabilising the Colour and Viscosity of an Organic-solvent-soluble Cellulose Ether. Hercules Powder Co. *BP* 734,968

Incorporation of a phenolic stabiliser and a resinous compound containing at least one epoxy group, e.g. ditertiary amylphenol and a polyglycidol, in an organic-solvent-soluble cellulose ether improves its colour and viscosity stability. This enables clear colourless or pastel-coloured moulded cellulose articles to be produced.

C. O. C.

Fibrous Materials having Improved Wet Strength. Johnson & Johnson. *BP* 734,725

A non-woven fibrous material, e.g. paper, contains as impregnant 0.1 to 8.0% (by wt. of the dry fibrous material) free acid cellulose glycollic or hydroxypropionic acid ether having in either case a degree of polymerisation indicated by a viscosity of a 0.5% solution of the free acid in 0.5 *N*-NaOH at 25.5°C. of at least 5.7 sec. (Ostwald-Fenske) compared with a zero pipette reading of 5 sec. for a 0.5 *N*-NaOH solution at 25.5°C. The ether has a degree of neutralisation > 50% and after treatment of the fibrous material is heated at > 175°F. long enough to make it water-insoluble. The degree of substitution may be above 0.25 and the insolubilising temperature above 245° or 300°F.

S. V. S.

Bleaching Cellulose Esters with Chlorites (VII p. 686).

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Pores in Collagen and Leather. R. R. Stromberg and M. Swerdlov. *J. Amer. Leather Chem. Assoc.*, 50, 336-354 (July 1955).

The approx. size and location of pores in collagen are shown. The pore-size range of 100-20,000 Å. radius has been investigated by means of a mercury pressure porosimeter, of which there is a diagram; the experimental method is given in full. Pore-size distribution curves of a number of leathers and collagenous tissues indicate that a significant pore volume is associated with pores < 1000 Å. in radius. Electron micrographs (four are reproduced) of collagen fibrils obtained from Hg-impregnated kangaroo tail tendon show periodic arrangements of opaque areas; these are interpreted as pore sites of ca. 150 Å. radius, within single fibrils. The geometry of swollen collagen fibrils, and of Hg-impregnated fibrils, suggests a helical configuration of the sub-fibrillar elements. The technological implications of these findings are discussed briefly. The tanning process probably does not appreciably alter the distribution of pore radii within the range 500-20,000 Å. Pores of the order of 150 Å. radius may provide paths for the transmission of water vapour, in addition to those provided by the porous fibrous

network as it is usually conceived. This may also have a direct bearing upon the profound changes that occur in the physical properties of leather when it is penetrated by water. The extent of impregnation by polymers, and therefore the properties of the finished product, may be better controlled if basic information is available regarding the pore structure of the leather and the physical structure of the impregnant.

J. W. D.

Structure and Deformation of Collagen Fibres. I—Plastic and Elastic Deformations under Pressure. A. J. Cruise. *J. Soc. Leather Trades Chem.*, 39, 252–262 (Aug. 1955).

Measurements of the elastic and plastic response of individual fibres can be correlated with the intrinsic structure of the fibres, with the type of tanning used, and with the action of solvents, etc. Qualitative work shows that the separation of fibres under pressure is closely dependent upon their state of hydration, and that the rate of loss of moisture may govern fibre identity upon drying. Excessive drying temp. or rates are associated with changes in the array of fibres in the dried bundle, and the surface appearance of the fibres can be emphasised by interferometry. Morphological variations are associated with marked differences in behaviour under pressure, and with markedly different "end-products", i.e. fragments or fibrils which assume characteristic shapes. There are 9 photomicrographs, and numerous graphs and diagrams.

J. W. D.

Nature of Acid Groups present in Chrome-tanned Collagen and the Pyridine Neutralisation Method of determining Complex-bound Acid. P. H. Rao. *J. Soc. Leather Trades Chem.*, 39, 217–224 (July 1955).

The acidity of the chromium complex fixed by collagen from 67% acid soln. of Cr sulphate is 33%, the rest of the sulphate being fixed ionically, irrespective of the time of tanning and the amount of Cr fixed. Drying the leather does not affect the acidity of the Cr complex. An attempt is made to calculate the proportion of uni- and bi-functionally fixed Cr from the results obtained by the diffusion neutralisation method, with a few assumptions; good agreement obtains with the values arrived at by Gustavson for gelatin (*J. Amer. Leather Chem. Assoc.*, 48, 575 (1953)). Chromium chloride and sulphate in 67% acid soln. seem to be fixed by collagen in the same way, probably with 33% acidity of the complex, but the latter gives the more stable complex.

J. W. D.

Studies in Chrome Tanning—Neutralisation of Chrome-tanned Leather. Y. Nayudamma and R. Theis. *J. Soc. Leather Trades Chem.*, 39, 239–251 (Aug. 1955).

Up to pH 4, protein-bound acid is preferentially titrated to chrome-bound acids. At pH 4.0, the chrome complex is stable; but at higher pH, the anion of the neutralising alkali penetrates the chrome complex, displacing the sulphate. Evidence is adduced in support of the view that in chrome tanning the Cr reacts with COOH groups of the protein, and not the NH₂ groups. A further modification of Gustavson's method (*J. Amer. Leather Chem. Assoc.*, 21, 559 (1926); *ibid.*, 22, 60 (1927)) is proposed for the correct determination of protein-bound acid.

J. W. D.

Paper Chromatography of the Non-tannins of Tanning Extracts. L. D. Pozzo. *Cuoio, pelli, mat. concianti*, 31, 71–82 (1955); *Chem. Abs.*, 49, 9947 (25 Aug. 1955).

Using aq. phenol as the solvent and aniline phthalate, p-anisidine and naphthoresorcinol to develop the spots on the paper the following were found to contain *inter alia*: Sumac extract—uronic acids, dehydroascorbic acid, glucose and arabinose; chestnut extract—dehydroascorbic acid, glucose, arabinose, xylose, galactose, fructose and arabinose; quebracho extract—glucose, arabinose, xylose, galactose, fructose and arabinose; wattle extract—uronic acids, glucose, arabinose and arabinose; gambier—ascorbic acid, glucose and arabinose; red spruce bark extract—uronic acids, glucose, arabinose, arabinose and ascorbic acid; green oak extract—glucose and arabinose.

C. O. C.

Cleaning of Furs in Sawdust. R. Vrbacký and V. Vrbacký. *Českoslov. kůžářství*, 3, 115–9 (1953); *Chem. Abs.*, 49, 7879 (10 June 1955).

Beech sawdust is the best, oak is also good but its tannin content colours the flesh side of the furs. Spruce sawdust is unsuitable. To differentiate between these three sawdusts boil a 5 g. sample for 10 min. in 150 ml. water filter to 50 ml., add 10 ml. 40% formaldehyde and 5 ml. conc. HCl and heat for 30 min. Filter, add 10 ml. of the filtrate to 1 ml. 2% FeCl₃ and 5 g. sodium acetate. When boiled with formaldehyde spruce gives no ppt., beech gives a fine brown ppt. and oak a pink ppt. and on adding FeCl₃ the colours produced are light brown, reddish brown and dark blue respectively. The moisture and/or the coarser the sawdust, the longer is required for cleaning.

C. O. C.

Peptides isolated from a Partial Hydrolysate of Steer Hide Collagen. II—Evidence for the Prolyl-Hydroxyproline Linkage in Collagen. T. D. Kroner, W. Tabroff, and J. J. McGarr. *J. Amer. Chem. Soc.*, 77, 3356–9 (20 June 1955).

Existence in collagen of a linkage between residues of proline and hydroxyproline has been established by isolation and determination of gly-pro-hydro and gly-(hydro, pro)-gly as the dinitrophenyl derivatives from a partial acid hydrolysate of steer hide collagen. Because of this the sequence gly-pro-hydro should be considered in any proposed structure of collagen. Twenty-eight peptides are reported for the first time and only 11 peptides out of the total 48 (including 17 tripeptides and one tetrapeptide) agree with the sequence -P-G-R- (prolyl or hydroxyprolyl-glycyl-an amino acid residue. Astbury, *J. Intern. Leather Tr. Chem.*, 24, 69 (1940)). The data obtained support the view that the repetitive sequence -P-G-R- is not an essential requirement for the structure of collagen.

C. O. C.

Structure of Polyglycine. C. H. Bamford, L. Brown, E. M. Cant, A. Elliott, W. E. Hanby, and B. R. Malcolm. *Nature*, 176, 396–397 (27 Aug. 1955).

Polyglycine I, prepared by casting from dichloroacetic acid or trifluoroacetic acid, and polyglycine II, prepared by casting from aqueous lithium bromide solutions, have different infrared spectra. Polyglycine I is a typical β -polypeptide. Consideration of the method of preparation and spectrum of polyglycine II shows that the configuration is not that of an α -helix. An intrachain hydrogen-bonded structure of folded molecules is suggested.

W. R. M.

PATENT

Removing Hair and Wool from Animal Hides and Skins. H. Zalcman. *BP 735,783*

Effective dewooling, with minimum damage to pelt or hair, is carried out using an aqueous paint substantially free from monovalent cations comprising water, a source of OH ions selected from a group comprising the oxides and hydroxides of Ca, Sr, and Ba, and a source of hydro-sulphide ions selected from a group comprising the sulphides and hydrosulphides of Ca, Mg, Sr, Al and Ba. The hydrosulphide ion content should be within 2–23.5% calculated as calcium hydrosulphide, and the pH 12.07–12.3. In one example the composition is (by wt.): 7.5% Ca(HS)₂, 15% CaO, 11% Ba(OH)₂, and 67% H₂O.

J. W. B.

Permanent Protection of Textiles against Microbiological Attack (X p. 690).

XIII—RUBBER; RESINS; PLASTICS

Variation of Elastic Properties of High Polymers with Orientation and Degree of Crystallinity. F. H. Müller. *Kolloid-Z.*, 142, 165–166 (July 1955).

Measurements of elastic moduli of foils permit the differentiation of degrees of orientation and directions of orientation. Elastic properties are determined from studies of the velocity of sound waves of 10 kilocycles frequency, corresponding to wavelengths of 10–50 cm. Damping also is estimated. Values of the elastic modulus and damping for polyethylene, oriented polystyrene, and non-oriented and oriented polyurethane are compared with those for certain metals. Values of the elastic modulus are much less than those for metals, and increase with

orientation. Differences are observed between unstretched polyurethan under fresh and stored conditions, and are ascribed to differences in crystallinity. W. R. M.

Dye-sensitized Photopolymerisation of Vinyl Compounds. Polymerisation of Styrene by Cyanine Dyes—I and II. M. Koizumi and A. Watanabe. *Bull. Chem. Soc. Japan*, 28, 136-140; 141-146 (March 1955).

I—Direct interaction between excited dye and monomer initiates polymerisation. The quantum yields for photobleaching and for initiation of polymerisation are of the order of 10^{-5} . One dye molecule produces one or two kinetic chains.

II—Using pinacyanol iodide as sensitiser, the effects of temperature, dye concentration, and wavelength of light on the photopolymerisation of styrene in a styrene-butanol mixture have been studied. The number of initiation processes produced by one decomposed dye molecule is < 1 at 40°C . and about 2 at 60°C . It is concluded that the initiation of polymerisation and the photobleaching are not necessarily identical although closely related. It is suggested that addition of dye and illumination only increase the initiation process. Under favourable conditions the fragment of decomposed dye molecule is attached to the polymer, which can also initiate photochemical polymerisation. W. R. M.

Intrinsic Viscosity-Molecular Weight Relationship for Polyacrylonitrile. R. L. Cleland and W. H. Stockmayer. *J. Polymer Sci.*, 17, 473-477 (Aug. 1955).
Weight-average molecular weights and intrinsic viscosities in dimethylformamide at 25°C . have been measured for four unfractionated acrylonitrile polymers, covering the mol.wt. range 30,000-250,000. The results follow the equation—

$$[\eta] = 2.43 \times 10^{-4} M_w^{0.71}$$

W. R. M.

PATENTS

Paper-like Materials from Thermoplastic Synthetic Materials. C. F. Roser. *BP 735,175*

Paper-like products are made from fibres of thermoplastic plastics, the fibres being stuck together at individual places by pressure and heat, heat being supplied to one side of a mixture of fibres, while exerting a pressure on points or places distributed over the surface, and the opposite side cooled, the other side then being treated similarly, melting into an impermeable foil being avoided. The mixture may be supplied continuously as a web of fleece or felt, two or more being superimposed if desired. The fibre directions of the two webs should preferably be at right angles to each other. An aftertreatment with finely-dispersed plastic, followed by pressure to produce or retain a smooth surface, may be applied. The apparatus comprises heated rollers, endless bands of heat-conducting material, e.g. wire fabric for guiding the web and pressing it on the heated rollers, and a cold roller. A further set of a heated, a pressure and a cold roller may be provided. S. V. S.

Moulding Multi-coloured Rubber Articles. Firestone Tire & Rubber Co. *BP 735,322*

A method of obtaining a sharp and uniform line of junction between light and dark coloured parts of the articles. C. O. C.

Shaped Polyethylene Articles. Deutsche Gold-und Silberscheideanstalt. *BP 735,162*

Polyethylene particles which have been swollen with an organic solvent capable of dissolving polyethylene at high temperatures are fused together into a continuous mass below the softening point of the initial polyethylene and then formed into shaped articles. W. G. C.

Filler for Thermosetting Resins. American Viscose Corp. *BP 732,257*

Moulded articles of good toughness and impact strength are obtained from thermosetting resins in which the fillers include $> 10\%$ (on the resin) of filler prepared by impregnating cotton wadding or fabric with a solution of a cellulose ester, e.g. cellulose acetate, drying and grinding. E. C.

Polyester Epoxide Resins. General Electric Co. *BP 732,253*

Resins of good stability on storage are obtained by mixing for a limited time at $80-150^\circ\text{C}$. a carboxyl-terminated alkyd or polyester resin (Acid No. > 150) with a conventional epoxide resin in such ratio that there are 0.8-1.2 carboxyl groups per epoxide group. E. C.

Plastics Constructional Materials in Textile Fibre Plants (I p. 670).

Dyeing Polyacrylonitrile Threads or Films (VIII p. 688).

XIV—ANALYSIS; TESTING; APPARATUS

Temperature Measurement of Metallic Surfaces. Anon. *Z. ges. Textilind.*, 57, 1057 (20 Aug. 1955).

Temperature measurements of metallic surfaces with instruments is not always accurate or straightforward. Messrs. Merck of Darmstadt have brought out a collection of organic chemicals which melt at specified temperatures and cover the range $40-190^\circ\text{C}$. Use of these products provides a simple and reliable method of assessment of the temp. of metallic surfaces such as presses, mangles, calenders, cylinder driers, ironing and pressing machinery, steam pipes, etc. B. K.

Applications of Chromatography in Inorganic Analysis. J. Marcé Piera. *Afinidad*, 32, 47-55 (March-April 1955).

The use of base-exchange resins with radioactive indicators is discussed. S. R. C.

Continuous Paper Chromatography. J. Solms. *Helv. Chim. Acta*, 38, 1127-1133 (Aug. 1955).

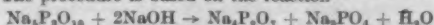
In the apparatus (in commercial production), the paper in the form of a cylinder is hung from a circular eluent trough and is rotated horizontally. The solution to be resolved is applied at a fixed point, and the components drip off separately into fixed receivers. H. E. N.

Colorimetric Determination of the Sulphate Ion. J. L. Lambert, S. K. Yasuda, and M. P. Grotheer. *Anal. Chem.*, 27, 800-801 (May 1955).

The SO_4^{2-} ion releases dye mol. from an insol. thorium borate-Amaranth dye reagent, in direct proportion to its concn. Determination of SO_4^{2-} is thus possible, in the range 0-400 p.p.m., by measurement of the dye concn. at 521 m μ . Other ions may interfere above limiting concn. which are listed, the most serious effects being those due to the PO_4^{3-} , F^- , and HCO_3^- ions; the last two may be eliminated by treatment with lanthanum nitrate, and a weak-acid ion-exchange column, respectively. J. W. D.

Determination of Soluble Ortho-, Pyro-, and Triphosphate in Presence of Each Other. L. E. Netherton, A. R. Wreath, and D. N. Bernhart. *Anal. Chem.*, 27, 860-861 (May 1955).

The procedure is based on the reaction—



The orthophosphate is first determined colorimetrically, by a modification of the molybdate method. A further sample of the soln. is hydrolysed with boiling NaOH, to effect the above reaction; the triphosphate may then be estimated from the change in orthophosphate content. Finally, the complete hydrolysis of a further sample by boiling acid yields orthophosphate alone; the total P content of the hydrolysate may thus be found, and the pyrophosphate estimated by difference. J. W. D.

Titration of Bases with Diphenyl Phosphate in some Aqueous and Non-aqueous Solvents. M. M. Davis and H. B. Hetzer. *Bur. Stand. J. Res.*, 54, 309-320 (June 1955).

A wide variety of bases may be successfully titrated with diphenyl phosphate (I) in solvents ranging from water to benzene, the end-point being detected potentiometrically in some cases, and in others by means of indicator dyes, some of which (Bromophthalein Magenta E, 5-phenyl-9-ethyl Nile Blue, and the anhydro-base of Tamarack Green and Victoria Blue B) have not previously been reported as acid-base indicators. Although I is of high acidic strength and of relatively high mol. wt., and is easily obtained and maintained in cryst. form, it cannot

yet be recommended as a standard for acidimetric work of the highest accuracy. It is not strong enough to replace HClO_4 for titrations in acetic acid. The behaviour of benzoic acid and I in water is different from that in methanol, changes in apparent acidic strength occurring (water appears to "level" the acidic strengths of different acids). Separate, sharp end-points may be obtained when a mixture of these two acids is titrated in a "non-leveling" solvent such as methanol or benzene-methanol.

J. W. D.

Mackey Test for Oleines. Institut Textile de France and Institut des Corps Gras. *Bull. Inst. Text. France*, (54), 55-62 (June 1955).

A precise specification for the Mackey test for the oxidizability of oleines is described on the basis of the results of interlaboratory trials.

J. C. F.

Rapid Method for the Estimation of Unsaturation of Fats and Oils by use of Hypochlorous Acid Reagent. S. Mukherjee. *J. Amer. Oil Chem. Soc.*, 32, 351-353 (June 1955).

A rapid method for the determination of unsaturation of oils and fats containing non-conjugated ethylene linkages has been developed, using hypochlorous acid as the reagent. The method has several advantages—the estimation is conducted with an aqueous solution of sodium hypochlorite reagent, and no oil solvent is required for the estimation. A reaction time of 4-5 minutes is recommended and the estimations are thus much more rapid than the customary Wijs or Hanus methods. The method compares favourably with the standard procedures currently employed for determination of unsaturation.

P. G. M.

Detergency Test Method using Radioactive Carbon Black Soil. J. W. Hensley, M. G. Kramer, R. D. Ring, and H. R. Suter. *J. Amer. Oil Chem. Soc.*, 32, 138-148 (March 1955).

Test methods for the evaluation of laundry detergents have been developed employing radioactive carbon black soil in cloth. Soil removal evaluations have been made with a number of different types of detergents using dry carbon soil, and a carbon-mineral oil combination. The two soils were found to give the same relative ratings of the detergents. Using a series of 12 detergents representing different types, soil removal and whiteness retention values determined with the tracer methods have been compared with conventional laboratory test methods. Soil removal evaluations differ considerably with the two methods, and non-correlating results can be related to the general types of detergent compositions involved. The tracer method appears to give evaluations free from some anomalies that have been recognised in the conventional laboratory tests.

P. G. M.

Colorimetric Method for the Quantitative Estimation of Ethylene Oxide Adducts. N. Schönfeldt. *Kolloid-Z.*, 142, 164 (July 1955).

The method is based on the affinity between ethylene oxide adducts and certain dyes. The dye is dissolved in an aqueous solution or dispersion of adduct, and an organic liquid, immiscible with the aqueous phase, is added to form a separate layer. A substance precipitating the adduct is added from a burette. The precipitate, which carries the dye with it, collects at the interface between the two layers. The addition of precipitant is continued until the aqueous layer is practically colourless. The amount of precipitant added is proportional to the quantity of adduct in solution, the exact relationship being obtained with solutions of known concentration. Azo dyes may be used. The amount of dye must not be such as to affect precipitation, and the dye should not be soluble in the organic liquid, which may be xylene or a paraffin hydrocarbon. Sulphuric and silicotungstic acids are suggested as precipitants.

W. R. M.

Detection of Surface-active Agents containing a Polyoxyethylene or Polyoxypropylene Group—by Pyrolysis with Phosphoric Acid. M. J. Rosen. *Anal. Chem.*, 27, 787-790 (May 1955).

Thermal decomp. of the polyoxyethylene linkage in the presence of H_3PO_4 yields acetaldehyde, which produces a blue colour with Na nitroprusside and diethanolamine. The polyoxypropylene group yields, under the same conditions, propionaldehyde, which produces an orange

colour. Ester, alkylaryl, sulphide, sulphonate, sulphate, amino, amido, and phosphate groups do not interfere. Under the conditions of the decomp., glycerides yield acrolein, which also produces a blue colour, even in the absence of the above groupings. The results of the application of the test to over 60 epd. are tabulated.

J. W. D.

Distinguishing Tannins from Polyhydric Phenols.

A. Michaluk. *Acta Polon. Pharm.*, 11, 263-271 (1954); *Chem. Abs.*, 49, 9948 (25 July 1955).

The following tests were used on a variety of 1% extracts of natural tanning agents (galls, catechu, etc.) and on gallic acid, quercetin and several polyhydric phenols—(a) FeCl_3 , (b) $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2$, (c) vanillin (acidified with HCl), (d) Mitchell reagent (0.1 g. FeSO_4 and 0.5 g. KNa tartrate per 100 ml. water), (e) 1% aq. gelatin, (f) bromine water, (g) 2% antipyrine soln., (h) 10% neutral Pb caprylate, (i) alkaline Pb caprylate, and (j) Braemer reagent (1 g. Na tungstate and 2 g. Na caprylate/10 ml. distilled water). The colour reactions given by (a) and (b) (blue, violet and green) cannot be used for specific determination of tannins; that of (c) (red or claret) shows presence of phloroglucinol in condensed tannins; (d) with a true tannin yields a black or violet ppt.; positive reaction with (e) indicates presence of tannins; condensed tannins are immediately precipitated by (f) and gallotannins after long standing; reaction with (g) tends to indicate presence of true tannins but cannot be taken as a positive test; (h) and (i) do not indicate presence of tannins as polyhydric phenols and quercetin are also precipitated; caprylic acid itself precipitates gallotannins; (j) is specific for precipitation of gallic acid and its derivatives.

C. O. C.

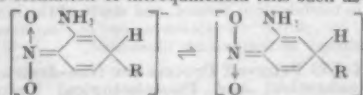
Detection of an Unsubstituted Para Position in Phenols. S. Soloway and A. Santoro. *Anal. Chem.*, 27, 798-800 (May 1955).

Phenols are readily oxidised to coloured products in ammoniacal soln. by the persulphate ion, in the presence of a catalytic amount of Ag^+ . Derivatives having a free para position are distinguished from others in that they yield blue or green dyes which are pH as well as redox indicators. Evidence is adduced which suggests that these dyes are indophenol derivatives. The nature of the catalysis by Ag^+ and the anomalous behaviour of a few phenols are discussed. p-Halogenated phenols give a positive reaction, and it is assumed that the halogen group migrates to another position in the ring, since the dyes are believed to be p-indophenol derivatives.

J. W. D.

Colour Reaction for Determination of Some Aromatic Nitro Compounds. C. C. Porter. *Anal. Chem.*, 27, 805-807 (May 1955).

Mononitro epd. produce orange, red, or purple colours when tetraethylammonium hydroxide is added to their soln. in dimethylformamide. The Janovsky reaction (in which acetone and NaOH are the reagents) may also be used to determine certain mononitro epd. The colours produced by 67 epd. are listed, together with the wavelength of max. absorption, and the absorption at that wavelength. It is believed that the colours are attributable to the formation of nitroquinonoid ions such as—



Fourteen of the epd. listed are amenable to quant. determination.

J. W. D.

Strength Assessment of Dyes. A. Wylezich. *Melliand Textilber.*, 36, 728-730 (July 1955).

In producing the standard from type, there are three possibilities—shading with other dyes, strengthening with concentrated dye, or reduction with the usual diluents, possibly adding alkylsilicones for dustless powders. The importance of the Hardy spectrophotometer is stressed, and the commercial tolerance of strength quoted as $\pm 5\%$.

S. R. C.

1-(2-Pyridylazo)-2-naphthol as a Possible Analytical Reagent. K. L. Cheng and R. H. Bray. *Anal. Chem.*, 27, 782-785 (May 1955).

The dye 1-(2-pyridylazo)-2-naphthol (I) forms coloured metal chelates, whose stability is sensitive to low pH. Ethylenediaminetetra-acetic acid (EDTA) prevents chelate

formation, and I may thus be used as an indicator in the complexometric titration of Zn, Cu, Cd, etc. with EDTA. The alkali and alkaline-earth metals do not form coloured chelates, and thus do not interfere. Fluoride and citrate ions prevent Fe, Bi, Pb, and Th from reacting with I, whilst in the presence of CN^- , only Bi and Pb react.

J. W. D.

Chromatography of Dyes on Circular Filter Papers.

J. Siedlecka. *Roczniki Państwowego Zakładu Hig.*, **2**, 33-42 (1951); *Chem. Abs.*, **49**, 9932 (25 July 1955).

Mixtures of dyes were separated chromatographically on circular filter papers (Schleicher & Schüll Ambocceptor Paper and E. & D. Filter Paper 6B) with distilled water, 1% NaCl and 1% NH_4OH as solvent. The following mixtures were separated: Orange I and Orange II, New Cocciniline and Ponceau 2R, Bordeaux 8 and Ponceau 3R, Water Blue and Patent Blue, Tartrazine and Acid Yellow. Mixtures of Acid Yellow with Naphthol Yellow and Naphthol Yellow with Tartrazine could not be separated. The method was used for separating and identifying food dyes, e.g. in coloured candy.

C. O. C.

Karl Fischer Reagent for Determining Moisture in Pigments.

Deut. Farben-Z., **9**, 76-8 (1955); *Chem. Abs.*, **49**, 7867 (10 June 1955).

A weighed sample is shaken with anhydrous methyl alcohol, allowed to settle for 18 hr. and an aliquot of the alcohol withdrawn and titrated with Karl Fischer reagent against a blank of the original alcohol. The water thus determined is usually higher than that formed by drying the pigment at 120°C. for 5 hr. Moisture data are given for 165 commercial pigments. Several organic pigments are soluble in methyl alcohol and some others interfere with observation of the end point. There was considerable variation in the amount of moisture absorbed by 9 pigments exposed to the atmosphere for 1-20 days, natural pigments generally taking up more than synthetic pigments. The principal factor in the moisture content of pigments is the conditions during storage.

C. O. C.

Flow and the Critical Pigment Volume in the Paint Industry—Measurement of Oil Absorption by Pigments and Critical Pigment Volume.

G. Zeidler and G. Eberle. *Kolloid-Z.*, **139**, 115-120 (1954); *Chem. Abs.*, **49**, 7867 (10 June 1955).

A review of the rheological properties of paints. Photographs are given of typical patterns formed when tung, oiticica and isomerised linseed oils are treated with drops of concn. H_2SO_4 . Oil absorption of pigments and critical pigment value are measured by placing 10 g. pigment in a glazed mortar of 11.5 cm. internal diameter. Oil is added from a 5-c.c. microburette, at the rate of 1 drop/5 sec., and ground in with a pestle about 12.5 cm. long weighing 80 g. Oil is added until the paste formed sticks to the pestle and the mortar is clean. The minimum oil concentration is expressed in c.c. per 100 g. of pigment.

C. O. C.

Electron Microscopy in Textile Research.

P. Kassenbeck. *Textiltech.*, **20**, 531-540 (July 1955).

The methods discussed include an examination of the surface of fibres and the effect of different treatments resulting in modification thereof, and study of the internal structure by direct transmission or by impressions. Ultramicrotomy techniques are illustrated.

S. R. C.

The ROX-UG Print-off Process for Non-destructive Microchemical and Phosphotopical Tests of Surfaces.

H. Reumuth. *Melliand Textilber.*, **36**, 533-539 (June 1955).

A development of the original ROX process was used for detecting microchemical quantities on surfaces and for observing changes in the surfaces of woven goods, e.g. under abrasion. Illustrated.

S. R. C.

SVAH Abrasion Machine.

H. Kägi. *Textil-Rund.*, **10**, 296-301 (June 1955).

A description of the standard abrasion tester developed by the SVA (Swiss Research Institution).

J. C. F.

Standard Test for the Abrasion Resistance of Fabrics.

Schweizerische Normen-Vereinigung und Schweizerischer Verband für die Materialprüfungen der Technik. SNV 98 534. *Textil-Rund.*, **10**, 305-311 (June 1955).

Specification for abrasion resistance tests. The standard apparatus is the SVAH abrasion machine (see preceding abstract).

J. C. F.

Detection of Artificial Food Dyes—II.

J. Eisenbrand. *Deut. Lebensmittel-Rundschau.*, **50**, 248-250, 283-290 (1954); *Chem. Abs.*, **49**, 7452 (10 June 1955).

Detailed procedures for analysis of the 18 food dyes permitted in Germany.

C. O. C.

Cross-sectioning Slide and its Applications.

E. C. Jolliffe. *Text. Research J.*, **25**, 541-545 (June 1955).

The slide is $1 \times 3 \times 0.015$ in. and is made from matt-surfaced Plastocel (DuP), a carbon-impregnated cellulose acetate plastic. It is perforated with three parallel rows of 12 holes each, the holes being 0.035 in. in diameter and spaced $\frac{1}{4}$ in. apart. Sample preparation and method of sectioning are described for multiple and single strands. The use is discussed of the slide in fibre identification, determinations of filament number and denier, in photomicrography, and in the preparation of comparatively thick cross-sections.

W. P. M.

Differentiation of Jute and other Vegetable Fibres by Acetyl Content.

T. H. Soutar and M. Bryden. *J. Textile Inst.*, **46**, T 521-T 528 (Aug. 1955).

Acetyl contents of various bast and leaf fibres are determined by cold dilute alkaline hydrolysis, and show a close relation with hemicellulose contents. They offer a basis for differentiating fibre types; e.g. the upper limit for jute is 92 milli-equiv. of acetic acid and the lower limit for *Hibiscus* fibres is 98 milli-equiv. There is even a consistent difference between the acetyl content of *capsularis* and *olitorius* jute fibres, the former being appreciably higher. Acetyl determinations can also be used to ascertain whether or not known fibres have previously undergone alkaline treatment, which readily liberates acetyl groups.

J. W. B.

One-phase Solvent Mixtures for the Separation of Amino Acids.

T. L. Hardy, D. O. Holland, and J. H. C. Naylor. *Anal. Chem.*, **27**, 971-974 (June 1955).

The R_f values of 22 common, naturally occurring amino acids on filter paper have been determined using 55 different one-phase solvent mixtures. Promising combinations are selected, it being assumed that satisfactory separation of two amino acids is obtained by one-dimensional means if the difference in R_f values in a solvent mixture is 5 or greater; for satisfactory separation by two-dimensional means, the sum of the differences in R_f values in the two mixtures must be 7 or more. A method is described whereby 16 or a mixture of 18 amino acids may be identified rapidly on a single two-dimensional chromatogram; all 18 may be characterised on 4 separate one-dimensional chromatograms. Treatment of the chromatograms with cyclohexylamine or dicyclohexylamine, prior to development with ninhydrin, brings about striking variations in the usual colours, making it easier to identify individual amino acids. Closely related amides and peptides give colours which differ from those obtained with the parent amino acids.

J. W. D.

Wear Properties of Resin-finished Rayon Staple Fabrics.—A New Method of Laboratory Assessment.

L. F. H. Breens and T. H. Morton. *J.S.D.C.*, **71**, 513-524 (Sept. 1955).

An analysis of the problem of the prediction of premature breakdown of crease-resist-finished pure and blended rayon staple fabrics in heavy-duty wear shows that current methods of laboratory assessment, based on flat abrasion motions, are inadequate. A new type of testing machine, the BFT Mark III, determines resistance to flexing and to ball penetration; from these two machine factors a "duty factor" is derived which correlates very well with resistance to premature wear in a series of about sixty fabrics whose wear behaviour is known with certainty. Three different mechanisms of abrasive breakdown of woven fabrics are distinguished and related to measurable machine factors; two only of these, ball penetration and flex abrasion, are components of the duty factor. Authors.

Iodination of Wool (VI p. 684).

Estimation of Alkali Solubility of Silk Fibroin (VI p. 684).

Identification and Testing of Polyamide Fibres (VI p. 685).

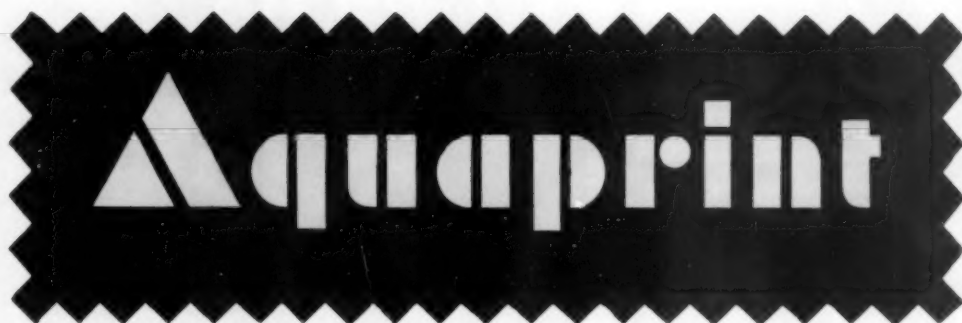
Microscopical Test for Characterisation of Different Radial Shells in Perlite (VI p. 685).

Quantitative X-Ray Determination of Amorphous Phase in Wood Pulp as related to Physical and Chemical Properties (XI p. 691).

Identification of Sawdusts (XII p. 693).

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FORTHCOMING MEETINGS OF THE SOCIETY—continued from page x

Friday, 17th February 1956

MANCHESTER SECTION. *A New Approach to High Temperature Dyeing*. S. N. Bradshaw, Esq., B.Sc. Textile Institute, 10 Blackfriars Street, Manchester 10. 6.30 p.m.

NORTHERN IRELAND SECTION. Dinner Dance. Details later, at Woodbourne House Hotel.

Monday, 20th February 1956

HUDDERSFIELD SECTION. *The Finishing of 'Terylene' and 'Terylene' Wool Blended Fabrics*. N. M. Mims, Esq., B.A., B.Sc. (Joint Lecture with the Huddersfield Textile Society). Large Hall, Technical College, Huddersfield. 7.30 p.m.

Thursday, 23rd February 1956

MIDLANDS SECTION. *The Finishing of 'Terylene' and 'Terylene' Wool Blends*. I. E. Haden, Esq., B.Sc., and B. I. Brooke, Esq., B.A. (Oxon.). Gas Theatre, Nottingham. 7 p.m.

NORTHERN IRELAND SECTION. *Film Evening*. Thompson's Restaurant, Donegall Place, Belfast. (Joint Meeting with the Textile Institute.) 7.30 p.m.

WEST RIDING SECTION. *Progress in the Cibalan Dye Range and their Application*. Clayton Dyestuffs Co. Ltd. Lecturer details later. The Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Friday, 2nd March 1956

LONDON SECTION. *Basic Mechanisms of Fading of Dyed Textiles*. H. B. Mann, Esq. (Courtaulds Ltd., Bocking). Royal Society Rooms, Burlington House, Piccadilly, London, W.1. 6 p.m.

BRADFORD JUNIOR BRANCH. Annual Dance. Connaught Rooms, Bradford.

Tuesday, 6th March 1956

LEEDS JUNIOR BRANCH. *The Textile Technologist in the Hosiery Trade*. F. M. Stevenson, Esq., M.Sc., J.P. (Messrs. Stevensons (Dyers) Ltd., Ambergate, Derbyshire). The Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, Leeds 2. 3.30 p.m.

Thursday, 8th March 1956

WEST RIDING SECTION. *A Commentary on the Use of Colour in Wool*. S. Morse-Brown, Esq. International Wool Secretariat. Ladies to be invited. The Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Friday, 9th March 1956

MIDLANDS SECTION. Midlands Section Dinner. Bull's Head Hotel, Loughborough.

Tuesday, 13th March 1956

SCOTTISH SECTION. *The Soiling of Synthetic Fibres*. G. G. Taylor, Esq., B.Sc., A.Inst.P. (Clayton Dyestuffs Co. Ltd.). St. Enoch's Hotel, Glasgow. 7 p.m.

BRADFORD JUNIOR BRANCH. *Some Aspects of Continuous Vat Dyeing*. Dr. R. J. Rallings (British Cotton Industries Research Association). Technical College, Bradford. 7.15 p.m.

Wednesday, 14th March 1956

NORTHERN IRELAND SECTION. *Various Methods of Stripping Vat, Azoic, Direct and Sulphur Dyes*. J. E. Riordan, Esq. (College of Technology, Belfast). *Effect of pH on the Activity of Bleaching Agents*. J. Boyd, Esq. (College of Technology, Belfast). Thompson's Restaurant, Donegall Place, Belfast. 7.30 p.m.

Tuesday, 20th March 1956

HUDDERSFIELD SECTION. *A New Approach to High Temperature Dyeing*. S. N. Bradshaw, Esq. (Courtaulds Ltd.). Co-operative Cafe, Huddersfield. 7.30 p.m.

Wednesday, 21st March 1956

MIDLANDS SECTION. *Fast Dyes on Cellulose Triacetate*. J. G. Graham, Esq., B.Sc. (Joint Meeting with British Association of Chemists). Midlands Hotel, Derby. 7 p.m.

Thursday, 22nd March 1956

MIDLANDS SECTION. *Dyeing Faults in the Carpet and Hosiery Industries*. G. H. Lister, Esq., B.Sc., Ph.D. (Joint Meeting with the Textile Institute). Carpet Trades Canteen, Kidderminster. 7 p.m.

WEST RIDING SECTION. Annual General Meeting. The Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Friday, 23rd March 1956

LONDON SECTION. *Dyeing of Narrow Fabrics—Cellulosic and New Fibres*. Dr. I. M. S. Walls (I.C.I. Ltd.). George Hotel, Luton. 7 p.m.

Saturday, 24th March 1956

NORTHERN IRELAND SECTION. Annual General Meeting and Dinner. Details later.

Tuesday, 10th April 1956

SCOTTISH SECTION. *Textile Invention and the Dry Cleaner*. A. J. Crockett, Esq., M.Sc., F.R.I.C. (Eastmans Dyers and Cleaners Ltd.). St. Enoch's Hotel, Glasgow. 7 p.m.

Friday, 20th April 1956

LONDON SECTION. Fourth London Lecture. *Colour in Fashion*. James Laver Esq. (Keeper of the Departments of Textiles and Engraving, Illustrations and Design, The Victoria and Albert Museum). Waldorf Hotel, London, W.C.2. 7 p.m. To be followed by a Dinner.

Saturday, 21st April 1956

BRADFORD JUNIOR BRANCH. Annual General Meeting. Technical College, Bradford. 10.15 a.m.

Wednesday, 25th April 1956

HUDDERSFIELD SECTION. Visit to Messrs. Stevensons Dyers Ltd., Ambergate, Derbyshire.

MIDLANDS SECTION. *A New Approach to High Temperature Dyeing*. S. N. Bradshaw, Esq. Kings Head Hotel, Loughborough. Preceded by the Annual General Meeting of the Section. 6.30 p.m.

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All inquiries relating to Advertisements in the Journal should be addressed to THE GENERAL SECRETARY, THE SOCIETY OF DYERS AND COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE.

Replies may be addressed Box —, THE SOCIETY OF DYERS AND COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE, where all communications relating to these Advertisements are treated in strict confidence.

SITUATIONS VACANT

The engagement of persons answering these advertisements must be made through a Local Office of the Ministry of Labour or a Scheduled Employment Agency if the applicant is a man aged 16-64 inclusive or a woman aged 18-59 inclusive unless he or she, or the employment, is excepted from the provisions of the Notification of Vacancies Order 1952.

CANADA. Technician wanted to assist the superintendent of the Dyeing Department of well established Synthetic Fibre Company situated in Quebec. Must have a sound knowledge of the dyeing and crease-resist finishing of Acetate/Viscose Staple Fabrics and preferably with some experience of Nylon and Terylene blends. First-class equipment and working conditions. Salary according to qualifications and experience and good prospects for man with ability. Group Insurance and Superannuation plan in operation. Reply in strict confidence giving full details of present position, education and personal background. Box V87

CHEMIST required for industrial plant engaged in the manufacture of all types of felt and needlelooms and the processing of raw materials including wool carbonising. Successful applicant will have opportunity to study wool processing in all its aspects with a view to eventual technical control. All applications to be marked "Chemist" and addressed to The Secretary, The Lancashire Felt Co. Ltd., P.O. Box 7, West Street, Denton, Manchester.

DEVELOPMENT CHEMIST. A young graduate with an honours degree or equivalent is required for organic process development. This progressive post offers unusually attractive opportunities for men of initiative and carries an initial salary of up to £800 p.a. Location will be with the Company's rapidly expanding production unit in East Lancashire.

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FIRM of Dyers and Finishers in the West Riding of Yorkshire require General Manager. Write in strict confidence giving full details of present position, salary, age, etc. Applicants must have first class technical and practical knowledge and experience. Box V83

JOHN WALTON OF GLOSSOP LTD (a subsidiary of Tootal Ltd.) require an assistant (male) for their new Textile Printing Laboratory. Applicants should have completed National Service and should possess a National Certificate or its equivalent. Consideration will be given to those about to qualify and facilities will be granted for further study. Laboratory experience, preferably in the textile finishing or dyestuffs industries, is essential. Applications should be addressed to the Secretary, Longdendale Works, Hollingworth, Manchester.

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REQUIRED by established dyestuff laboratory in Bradford, young man having completed Army service. Possibility of becoming technical representative. Pension Scheme, etc. Box V86

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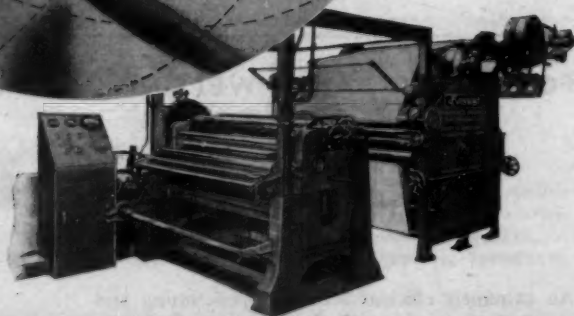
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